UCRL-PRES-433855

X-ray optics for the LCLS free-electron laser

Regina Soufli

Lawrence Livermore National Laboratory

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Contributors

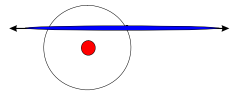
- LLNL: Sherry L. Baker, Mónica Fernández-Perea (LLNL and CSIC), Jeff C. Robinson, Stefan Hau-Riege, Tom J. McCarville, Anton Barty (now with CFEL), Michael J. Pivovaroff, Jay Ayers, Mark A. McKernan, Donn H. McMahon, Richard Bionta
- LBNL: Eric M. Gullikson, Phil Heimann
- SLAC: Peter Stefan, William Schlotter, Michael Rowen

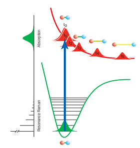
Regina Soufli
regina.soufli@llnl.gov

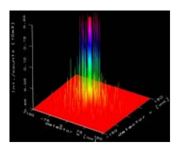
Linac Coherent Light Source (LCLS): the first x-ray Free-Electron Laser (FEL)

Courtesy: Jerry Hastings, SLAC/SSRL

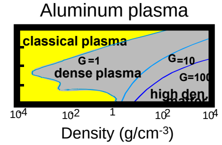
SLAC Report 611







Aluminum plasma



Atomic, Molecular and Optical science (AMO)

Soft X-Ray materials science (SXR)

Diffraction studies of stimulated dynamics: X-ray Pump-Probe (XPP)

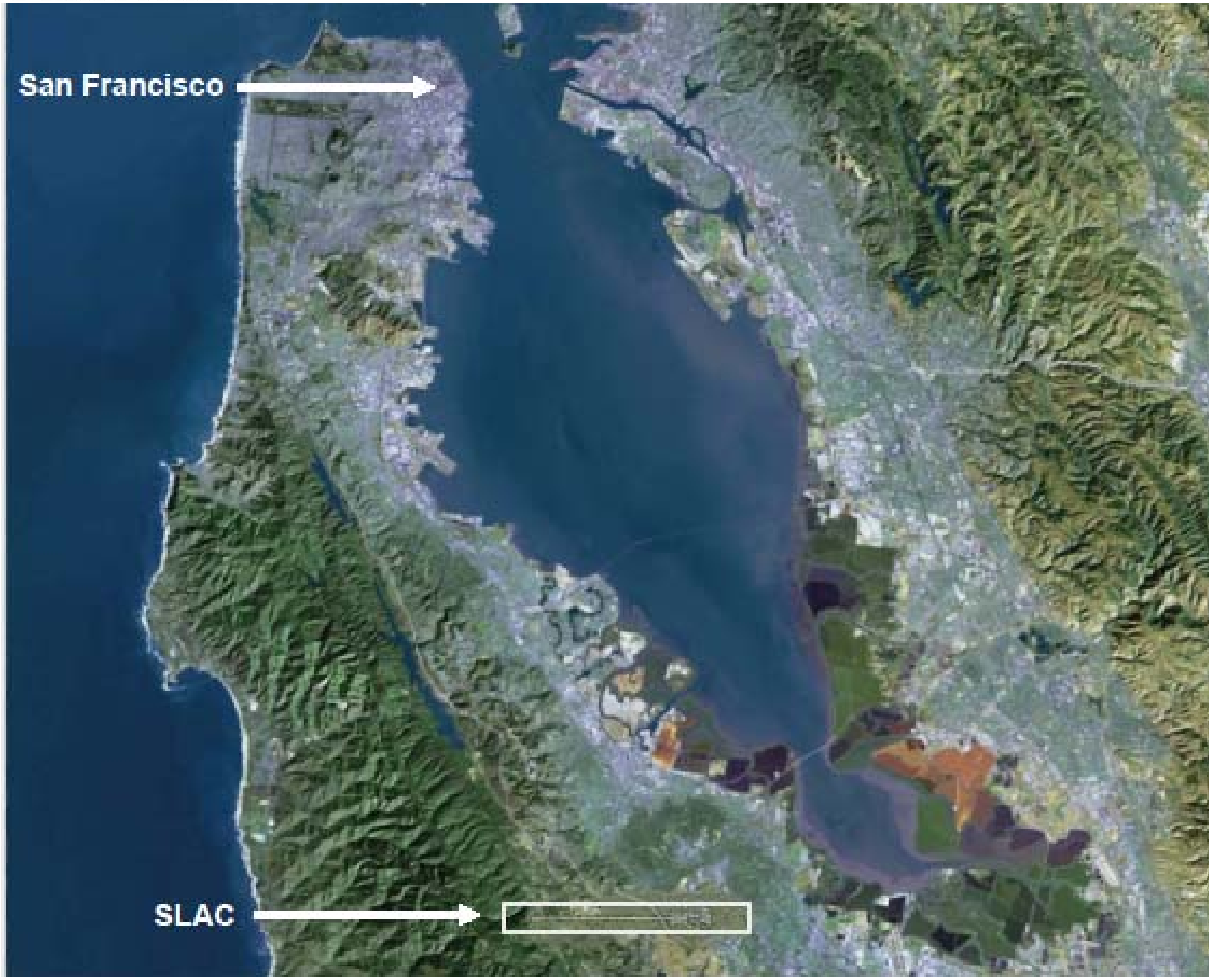
X-ray Photon Correlation Spectroscopy (XPCS)

Nano-particle and single molecule Coherent X-ray Imaging (CXI)

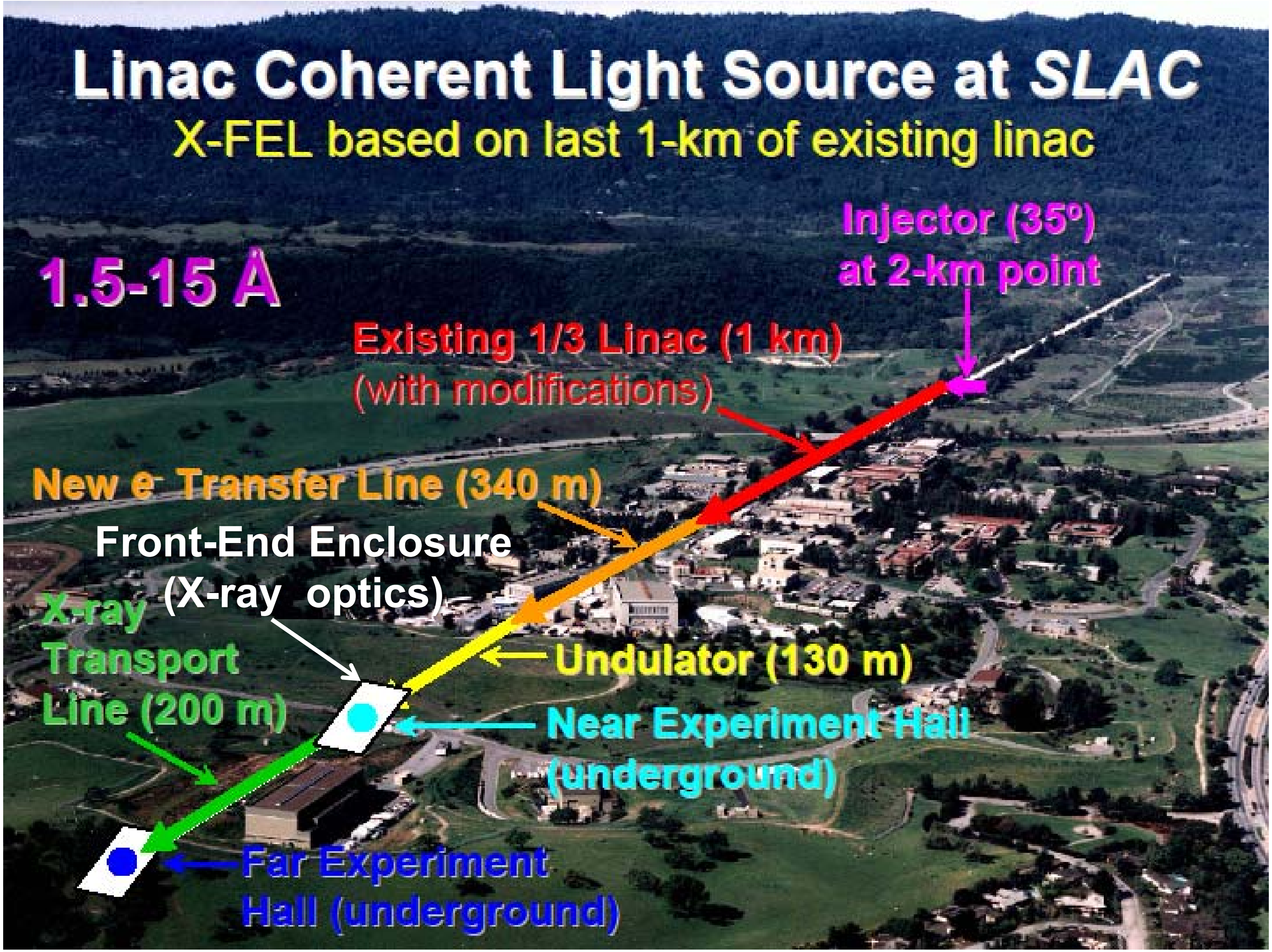
Materials under Extreme Conditions (MEC)

Regina Soufli
regina.soufli@llnl.gov

The LCLS is located at the SLAC National Accelerator Center in Menlo Park, California



Regina Soufli
regina.soufli@llnl.gov



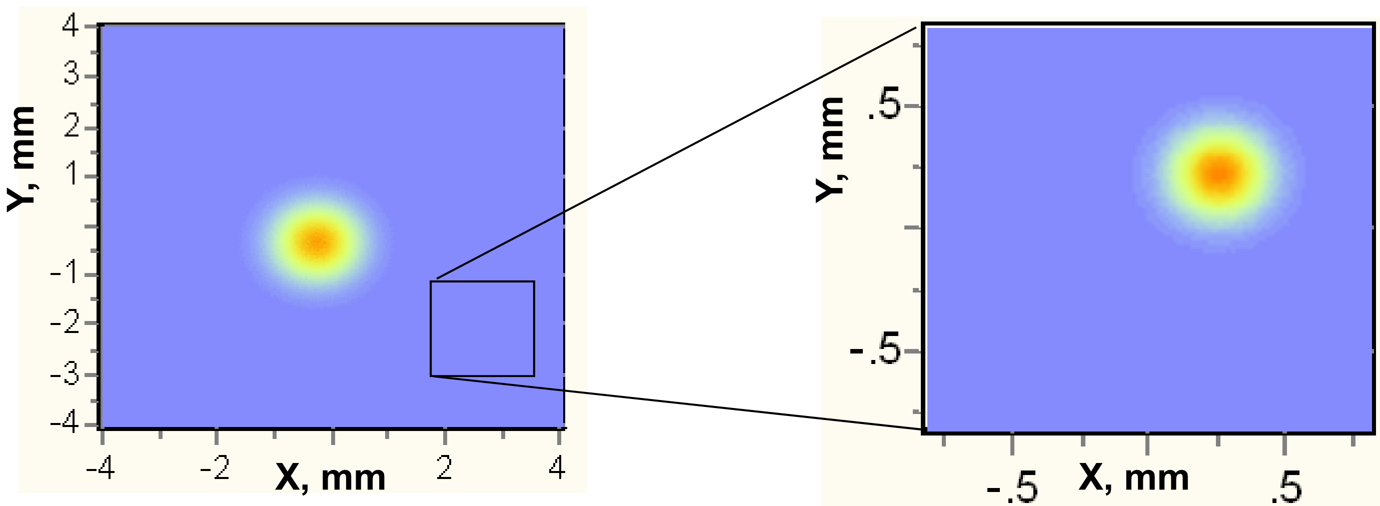
Courtesy: Sébastien Boutet (SLAC)

Regina Soufli
regina.soufli@llnl.gov

The FEL is small and bright in the Front-End Enclosure

1.5 nm Soft X-Ray FEL
(826 eV photon energy)

1.5 Å Hard X-Ray FEL
(8261 eV photon energy)



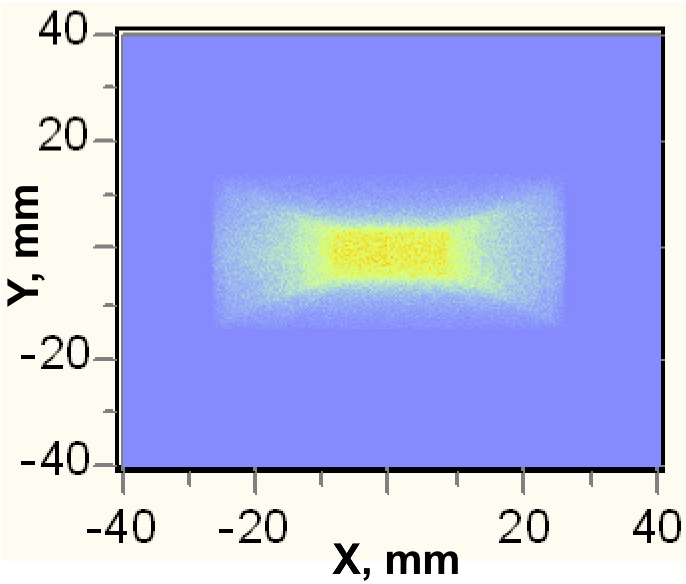
1.5 nm Soft X-Ray FEL:
1.9×10^{13} photons, total
2.5×10^{-3} J, total
0.2 J/cm^2, center
1.1 mm, FWHM
120 Hz, 137 fs pulses

1.5 Å Hard X-Ray FEL:
1.8×10^{12} photons, total
2.4×10^{-3} J, total
3.4 J/cm^2, center
160 mm, FWHM
120 Hz, 73 fs pulses

Regina Soufli
regina.soufli@llnl.gov

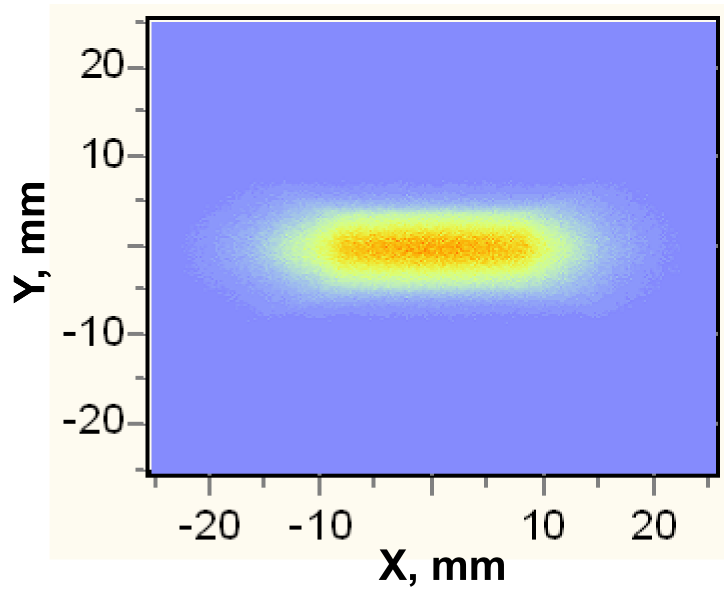
.. but is accompanied by spontaneous radiation

Soft X-Ray Spontaneous (4.5 GeV Linac K. E.)



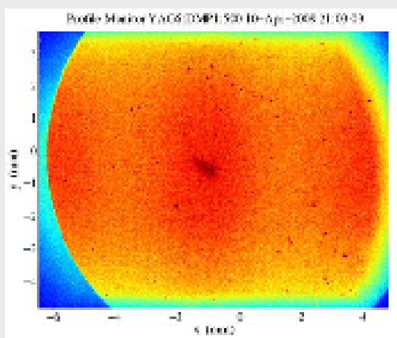
2.1×10^{11} photons, total
3.8×10^{-4} J, total
9.1×10^{-5} J/cm^2, center

Hard X-Ray Spontaneous (14.5 GeV Linac K. E.)

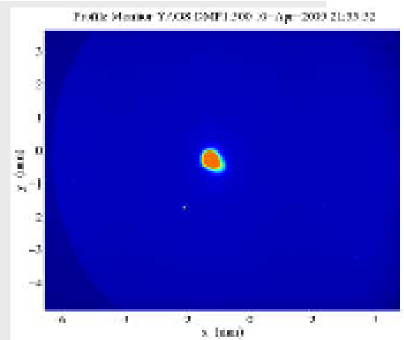


9.2×10^{11} photons, total
1.3×10^{-2} J, total
7.3×10^{-3} J/cm^2, center

Regina Soufli
regina.soufli@llnl.gov

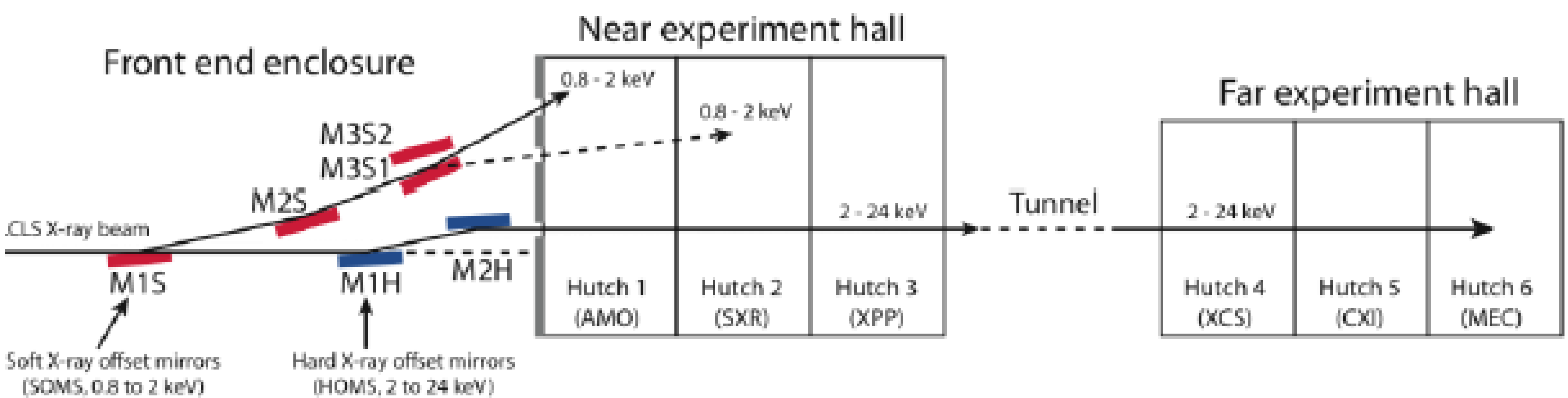


Spontaneous Beam



FEL beam

LLNL constructed the LCLS front-end enclosure x-ray optics and diagnostics, including the Soft X-ray and Hard X-ray Offset Mirror Systems (SOMS and HOMS). Also developed coatings and metrologies for x-ray mirrors and gratings for the AMO, SXR , CXI and MEC beamlines



Unique requirements for SOMS and HOMS:

- **Withstand instantaneous peak power of LCLS FEL (materials)**
- **Coherence/intensity preservation of LCLS wavefront (< 2 nm rms figure, 0.25 nm rms MSFR)**
- **Pointing stability and resolution (< 1 μrad for SOMS, 100 nrad for HOMS)**

Regina Soufli
regina.soufli@llnl.gov

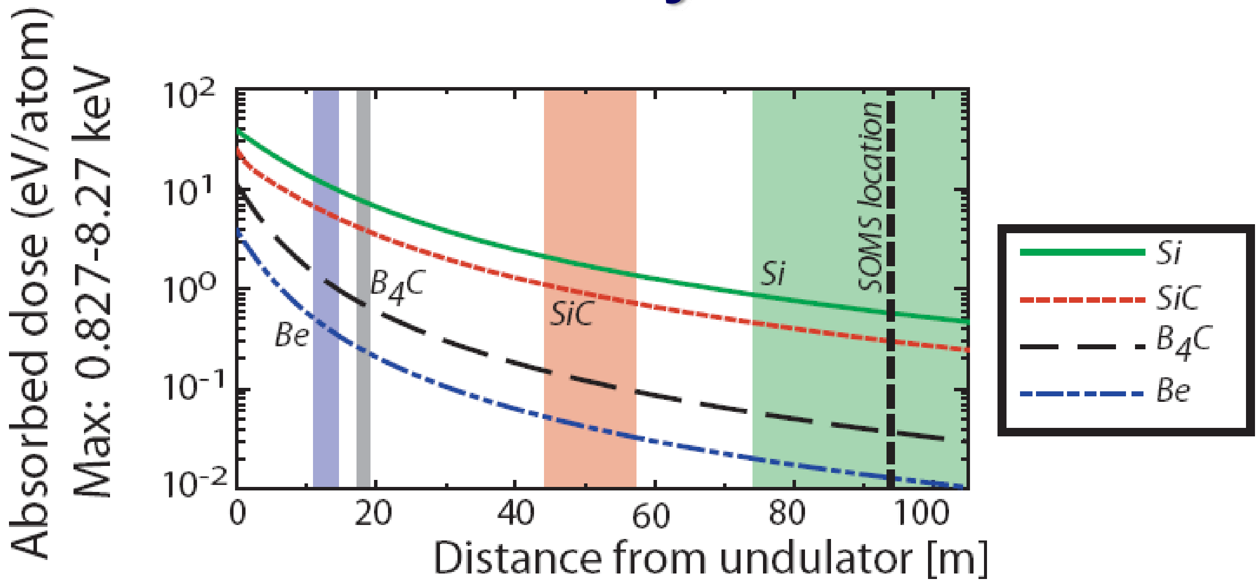
LCLS characteristics lead to novel considerations

Parameter	0.827 keV	8.27 keV
FEL pulse (rms)	137 fs	73 fs
FEL width (FWHM)	81 μm	60 μm
FEL divergence (FWHM)	8.1 μrad	1.1 μrad
FEL brightness [ph s^{-1} mm^{-2} $mrad^{-2}$ (0.1% bw)$^{-1}$]	0.28×10^{32}	15×10^{32}
Avg FEL power (at 120 Hz)	0.23 W	0.23 W
Avg Spontaneous power (at 120 Hz)	0.24 W	2.2 W

- Do not have traditional thermal issues associated with synchrotron mirrors
- Instead, concern is **instantaneous** damage
- Consider dose delivered eV/atom

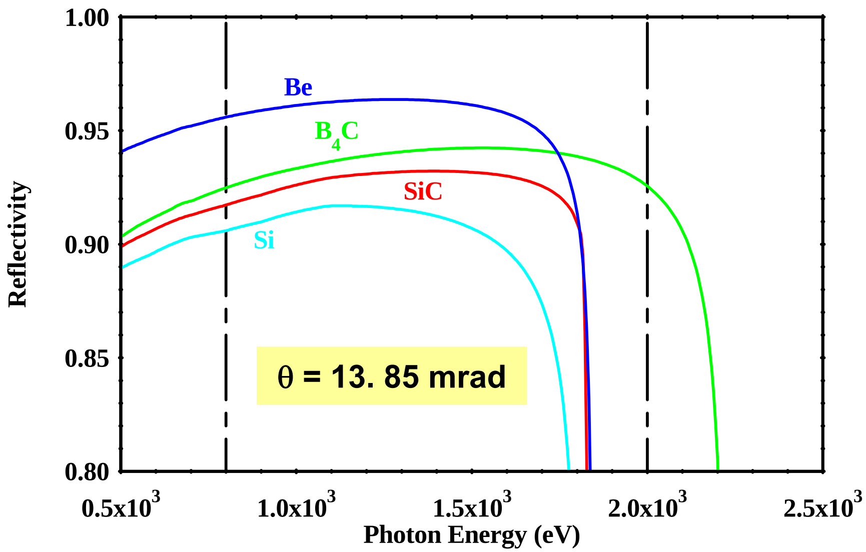
Regina Soufli
regina.soufli@llnl.gov

FEL beam dose considerations restrict the material choices for the LCLS x-ray mirrors

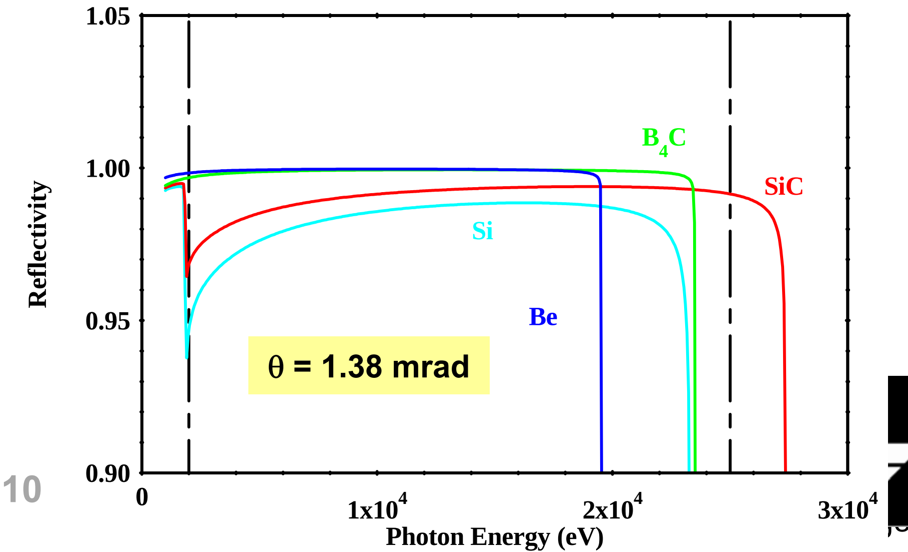


Vertical bands = range of distances over which the absorbed FEL dose will reach melting temperature, or melt each material

Soft x-ray region: $0.5 < h\nu < 2$ keV



Hard x-ray region: $2 < h\nu < 8\ (24.8)$ keV



Manufacturing approach for LCLS x-ray mirrors

- For LCLS soft x-ray mirrors: B_4C
  - Polish/figure B_4C monolithic mirror: infeasible
  - Procure Si substrate from commercial vendor, deposit 50-nm thick B_4C reflective coating at LLNL
- For LCLS hard x-ray mirrors: SiC:
  - Polish/figure SiC monolithic mirror: very challenging
  - Procure Si substrate from commercial vendor, deposit 50-nm thick SiC reflective coating at LLNL

Regina Soufli
regina.soufli@llnl.gov

Summary of LCLS x-ray mirror specifications

Error Category		Specification	Spatial Wavelength
Figure	Height Error	≤ 2.0 nm rms‡	1 mm to Clear Aperture†
	Slope Error	≤ 0.25 μrad rms	
Mid-Spatial Roughness		≤ 0.25 nm rms	2 μm to 1 mm
High-Spatial Roughness		≤ 0.4 nm rms	20 nm to 2 μm

† SOMS mirrors: Flat, planar, 250 × 30 × 50 mm^3, Clear Aperture = 175 × 10 mm^2

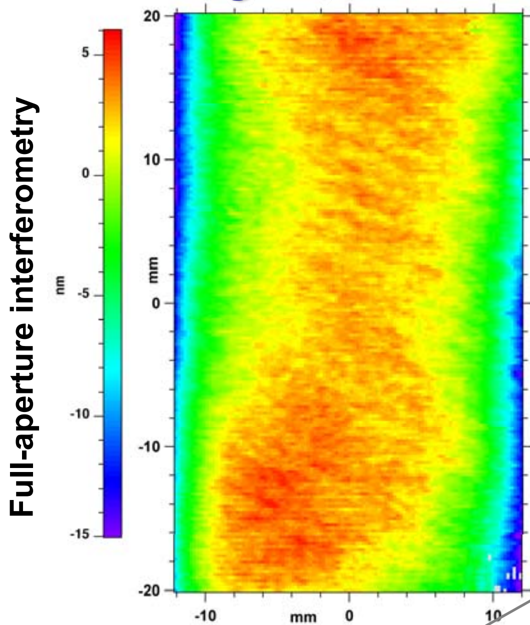
† HOMS mirrors: Flat, planar, 450 × 30 × 50 mm^3, Clear Aperture = 385 × 15 mm^2

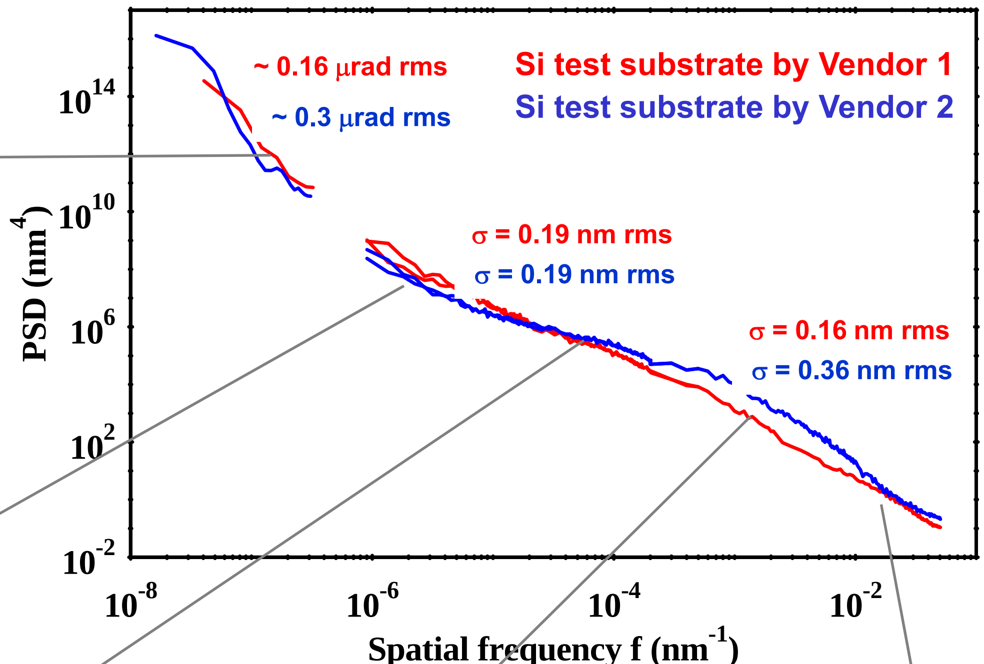
‡2 nm rms height error derived from Maréchal criterion: wavefront error < λ/14 rms

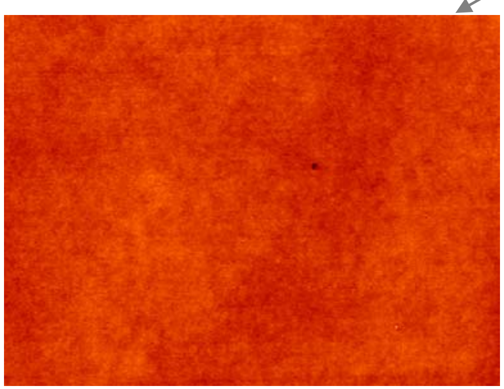
M. Pivovaroff, R. M. Bionta, T. J. Mccarville, R. Soufli, P. M. Stefan, "Soft X-ray mirrors for the Linac Coherent Light Source", Proc. SPIE 6705, 67050O (2007).

Regina Soufli
regina.soufli@llnl.gov

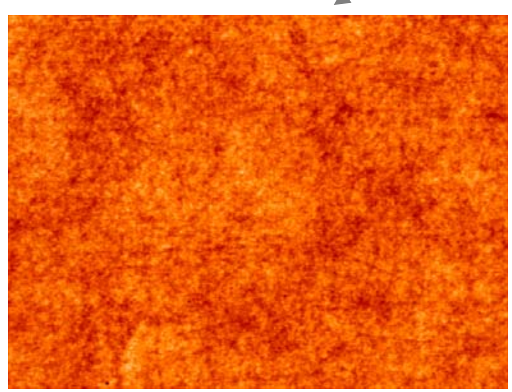
Precision surface metrology was performed at LLNL on the LCLS mirror substrates in the figure, mid- and high spatial frequency ranges



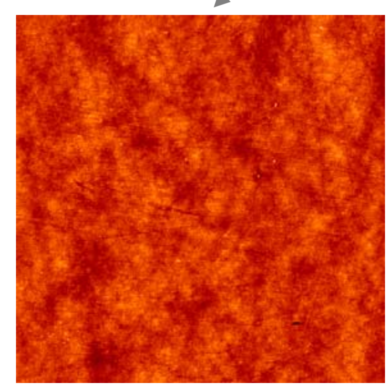




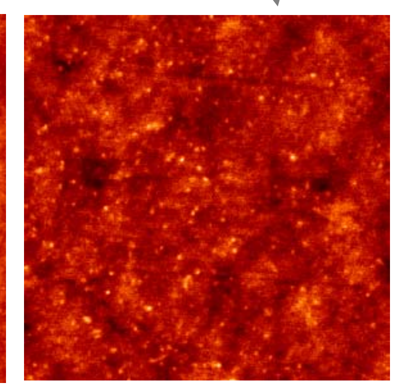
2.95 mm
Zygo 2x



0.37 mm
Zygo 20x



10 µm
AFM

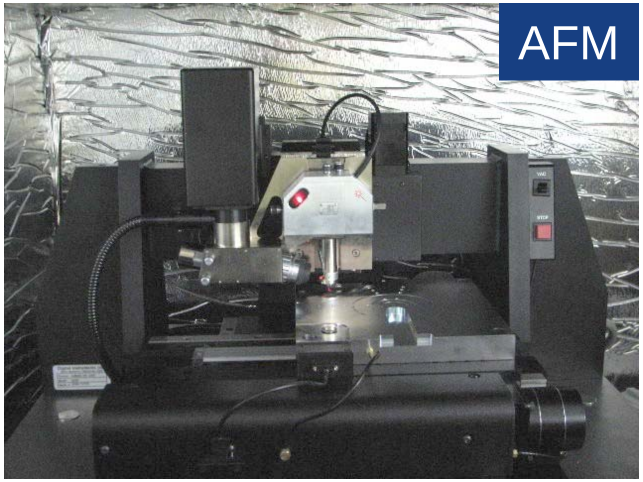


2 µm
AFM

Regina Soufli
regina.soufli@llnl.gov

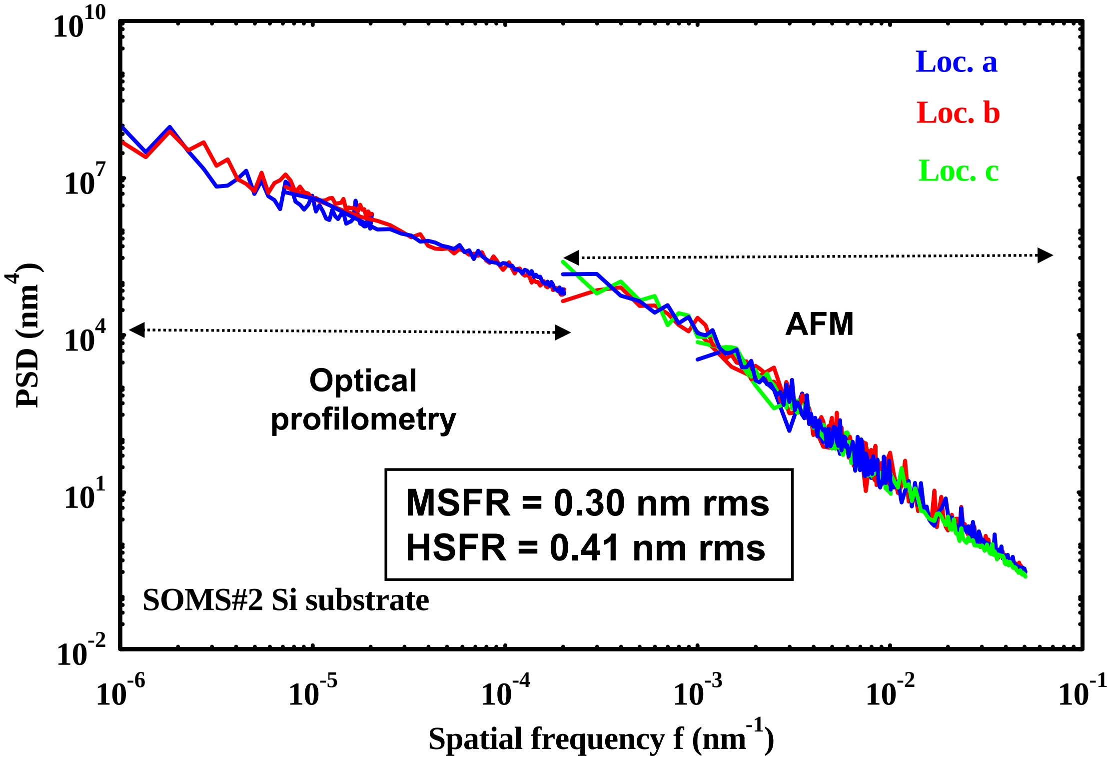
LLNL precision surface metrology lab

- *Digital Instruments Dimension 5000*™ Atomic Force Microscope (AFM) includes acoustic hood and vibration isolation. Noise level = 0.03 nm rms
- *Zygo NewView*™ Optical Profiling Microscope
- *LEO 1560* ™ Scanning Electron Microscope (SEM)



Regina Soufli
regina.soufli@llnl.gov

SOMS Si substrate metrology at LLNL



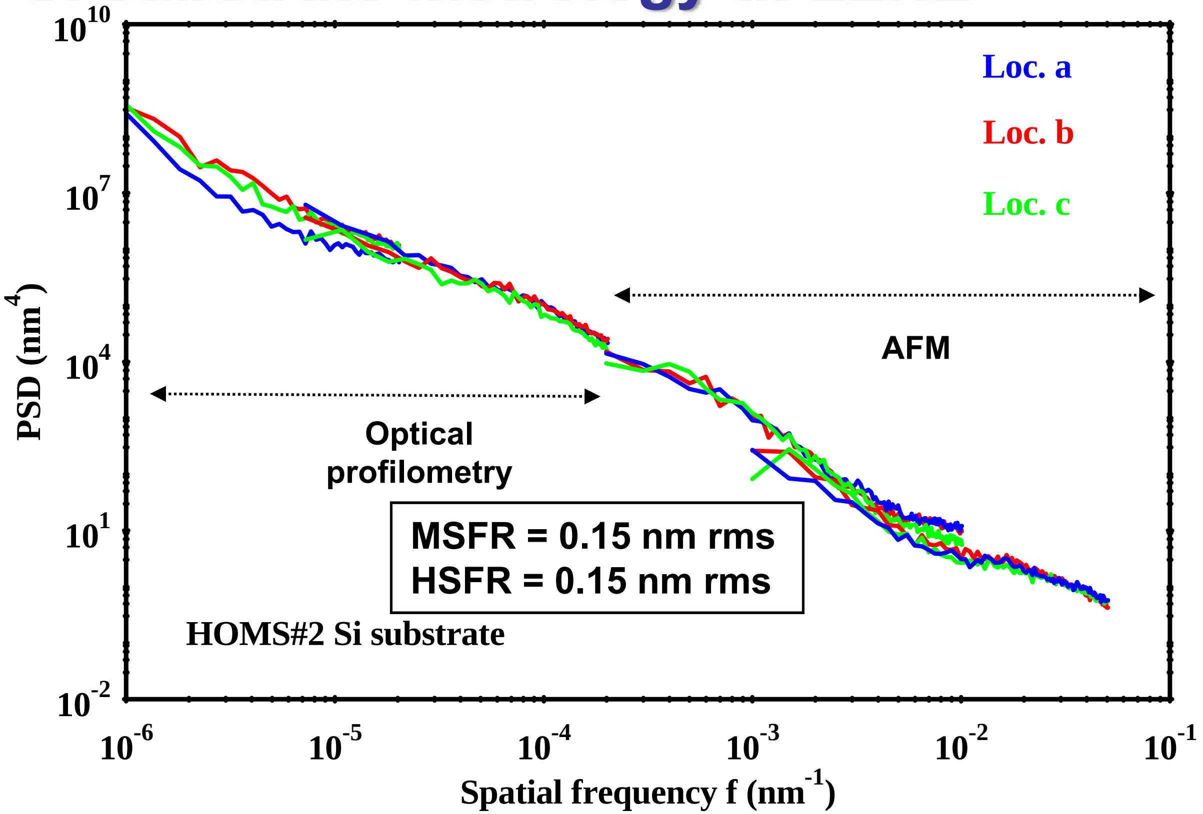
Mirror #	Figure (nm RMS)	Slope error (µrad RMS)
SN1	1.8	0.19
SN2	1.3	0.2
SN3	1.2	0.37
SN4	0.64	0.14
SN5 (spare)	1.4	0.37

Measured along central 200 mm

SOMS Si substrates manufactured by InSync (Albuquerque, New Mexico)

Regina Soufli
regina.soufli@llnl.gov

HOMS Si substrate metrology at LLNL



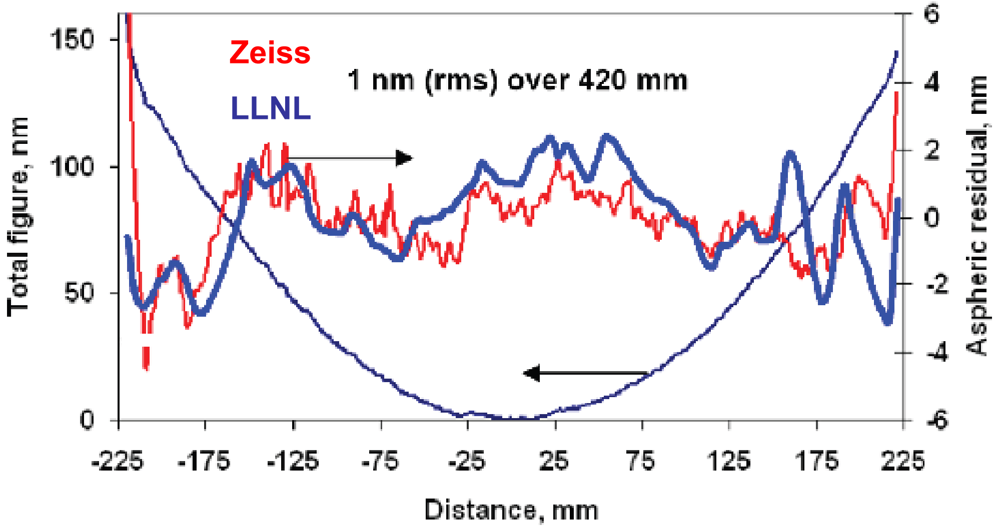
Mirror #	Figure (nm RMS)	Slope error (µrad RMS)
SN1 (blank)	2.4	0.27
SN2	1.0	0.27
SN3 (spare)	2.0	0.22
SN4 (spare)	1.5	0.23

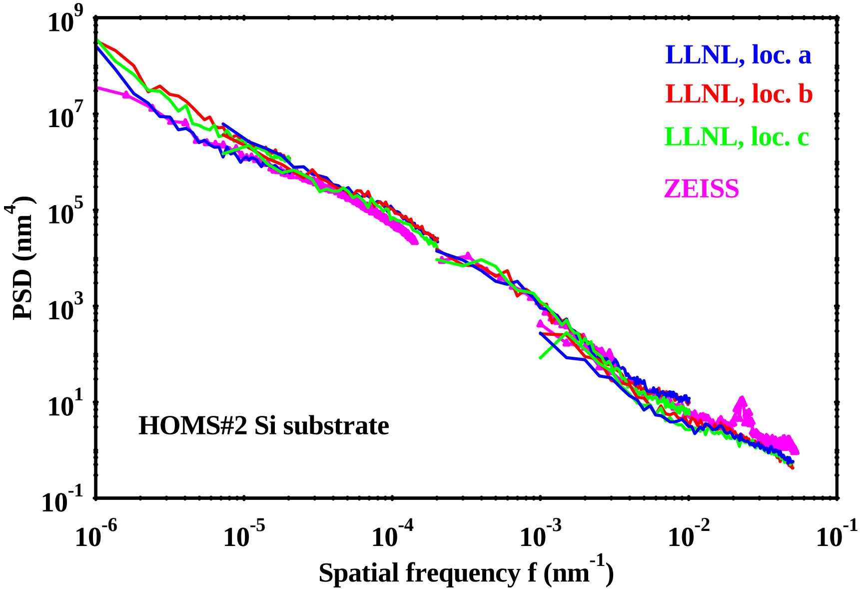
Measured along central 420 mm

HOMS Si substrates manufactured by Carl Zeiss Laser Optics (Oberkochen, Germany)

Regina Soufli
regina.soufli@llnl.gov

Recent comparison of LLNL and Zeiss metrology in the figure, mid- and high-spatial frequency ranges



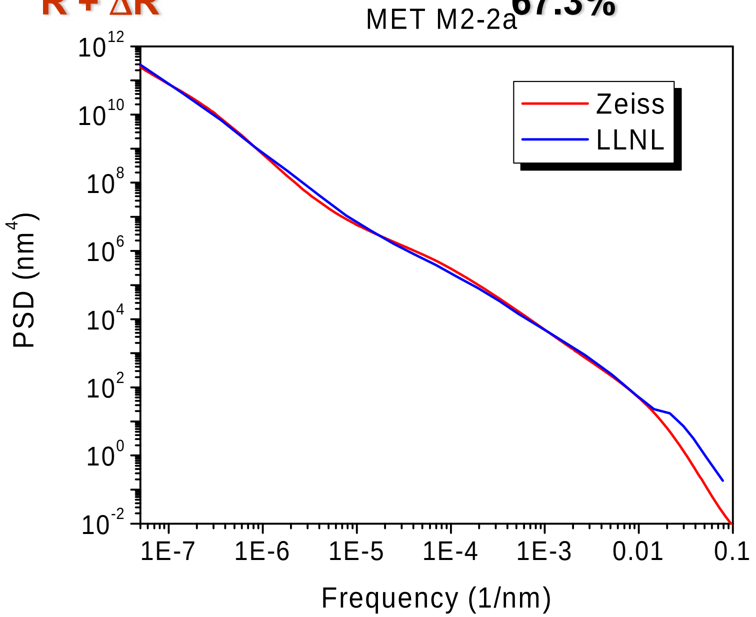


Zeiss measurements courtesy of Helge Thiess

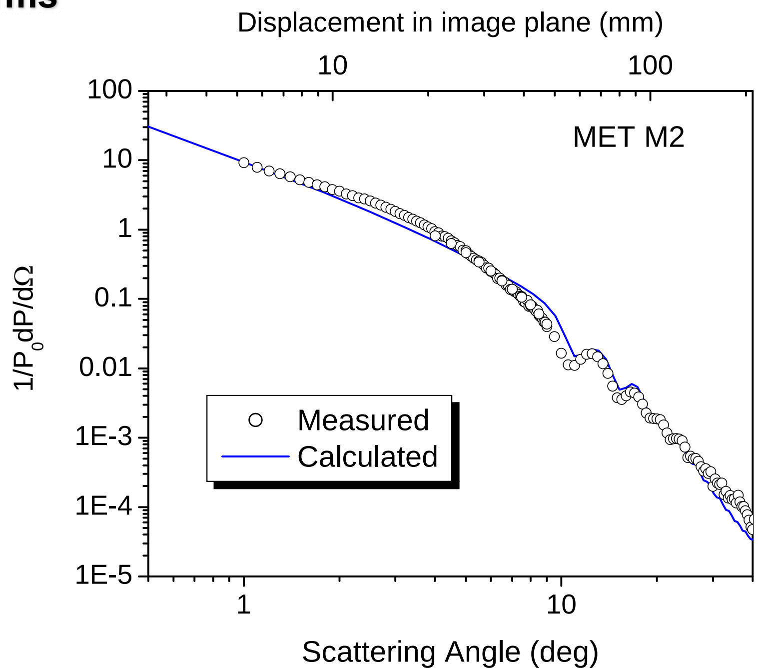
Regina Soufli
regina.soufli@llnl.gov

LLNL metrology validation with independent measurements and models

	MET 1 (primary)	MET 2 (secondary)
Substrate HSFR	0.37 nm rms	0.32 nm rms
Expected loss ΔR	6.1%	5.2%
Measured R	61.2%	62.4%
R + ΔR	67.3%	67.6%



LLNL / Zeiss metrology validation



LLNL metrology / scattering model / ALS scattering measurements validation

R. Soufli *et al.*, *Appl. Opt.* 46, 3736-3746 (2007)

D. G. Stearns, "Stochastic model for thin film growth and erosion," Appl. Phys. Lett. 62, 1745-7 (1993).
E. M. Gullikson, "Scattering from normal incidence EUV optics", Proc. SPIE 3331, 72-80 (1998).
D.G. Stearns *et al*, "Non-specular x-ray scattering in a multilayer-coated imaging system", J. App. Phys. 84, 1003-1028 (1998).

regina.soufli@llnl.gov

Reflective coating requirements for LCLS x-ray mirrors

- Figure: Coating should preserve the substrate figure specification of 0.25 μrad rms / 2 nm rms

  $\sigma_{total}^2 = \sigma_{sub}^2 + \sigma_{film}^2$

  => Coating is allowed to contribute < 1 nm rms figure error across clear aperture (175×10 mm^2 for soft x-ray mirrors, 385×5 mm^2 for hard x-ray mirrors)

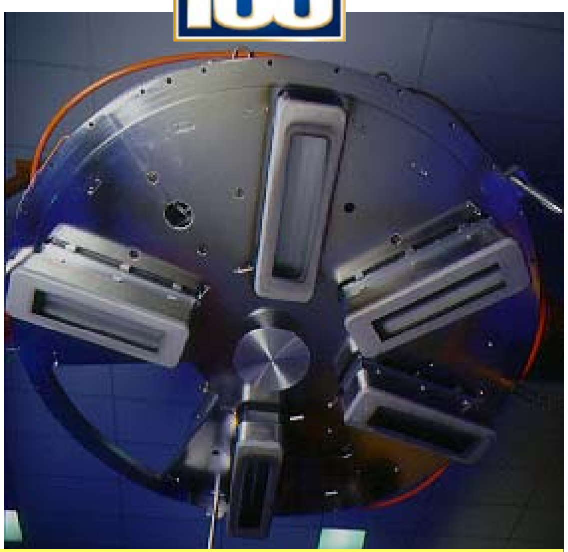
- Roughness: Coating should have low HSFR (will inherently replicate MSFR)

- Stress: Coating should have low stress (< 1GPa for ~50 nm coating thickness), to prevent figure deformation or delamination from Si substrate

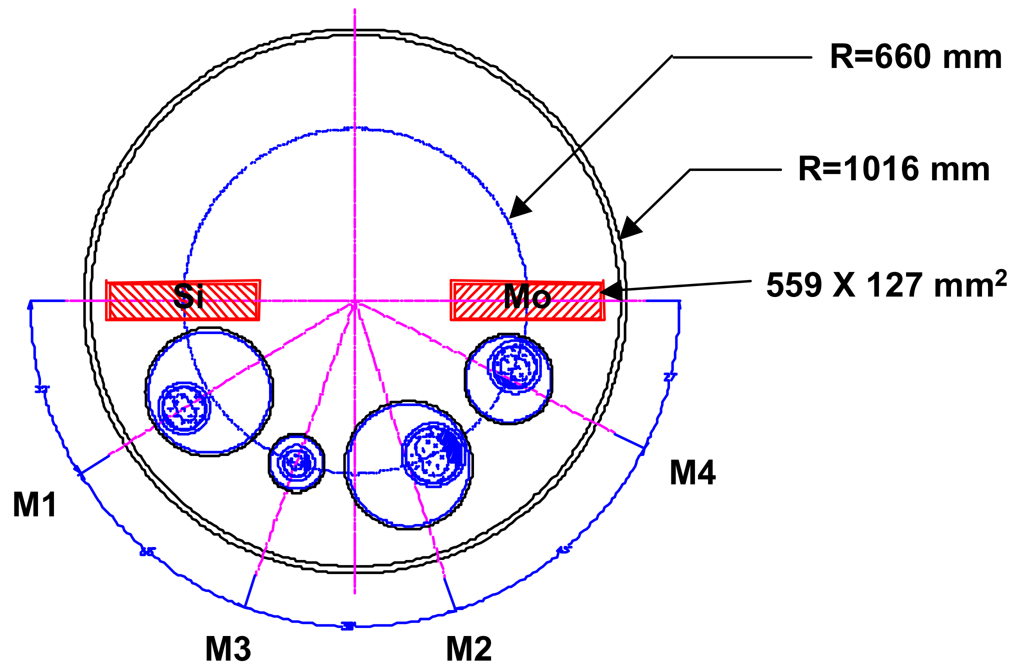
- Lifetime stability: Coating should be stable over time in ambient conditions, and under the operating conditions of LCLS x-ray mirror system

Regina Soufli
regina.soufli@llnl.gov

The LLNL DC-magnetron sputtering system is used to coat single-layer and multilayer optics



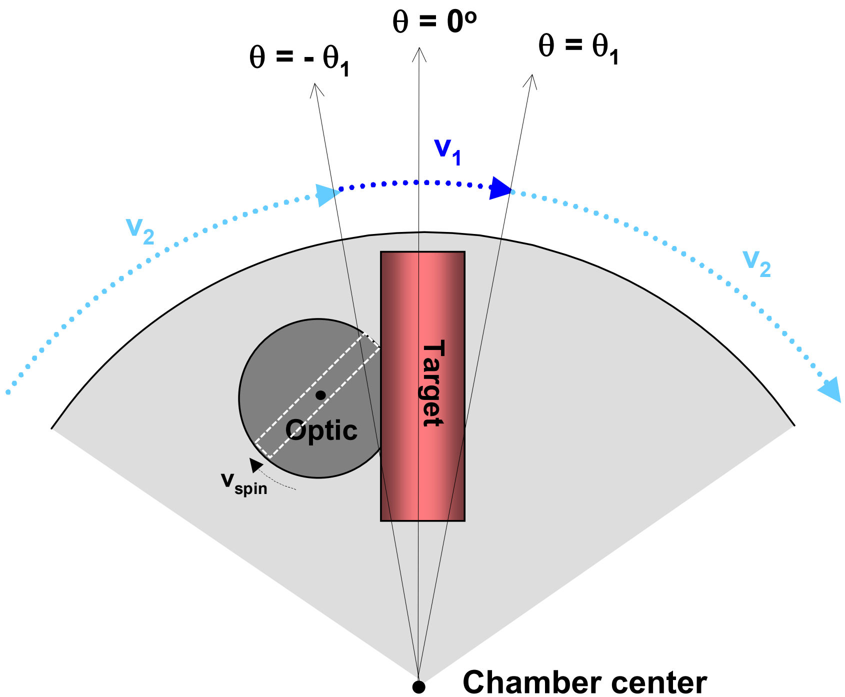
Underneath view of LLNL chamber lid with 5 sputtering targets



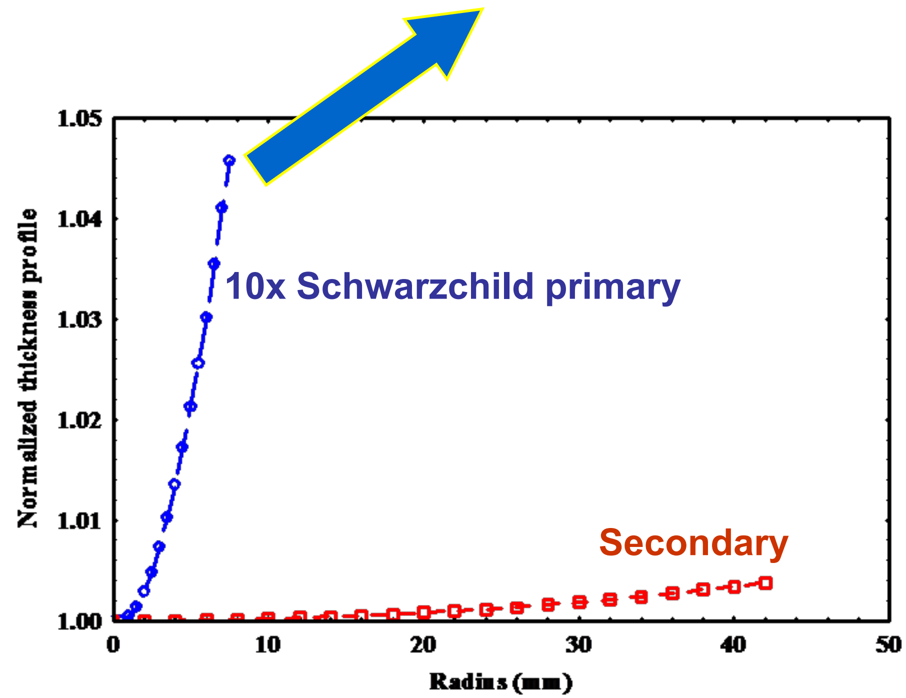
Can accommodate multiple mirrors as long as 450 mm into chamber without significant modifications to existing hardware

Regina Soufli
regina.soufli@llnl.gov

Velocity modulation is a rapidly converging method used for ultra-precise thin film thickness control



Achieved using velocity modulation. Until recently, such steep thickness gradients were believed to be possible only by positioning hardware masks over the optical substrate



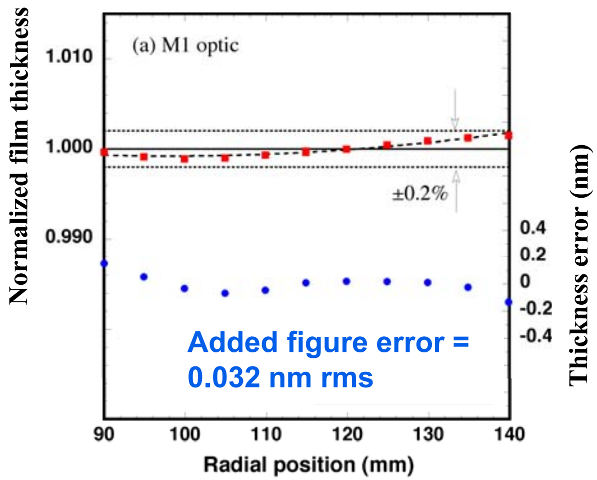
1. **Simulate deposition process**
2. **Select optimum (v_2/v_1, θ_1), to achieve desired thickness profile**
3. **Measure results on test optic and iterate**

R. Soufli, *et. al*, Appl. Opt. 46, 3736-3746 (2007).

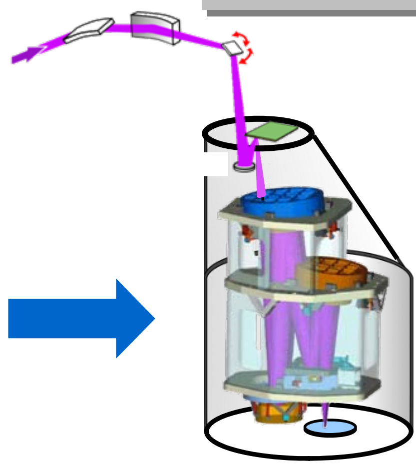
Regina Soufli
regina.soufli@llnl.gov

We have developed multilayer projection optics with sub-diffraction-limited performance during the EUVL program

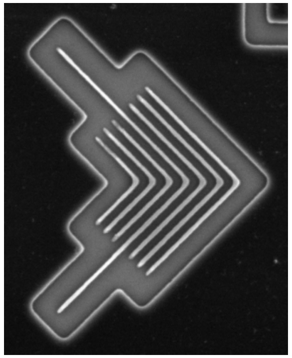
M1 mirror, PO Box 2



SES at ALS

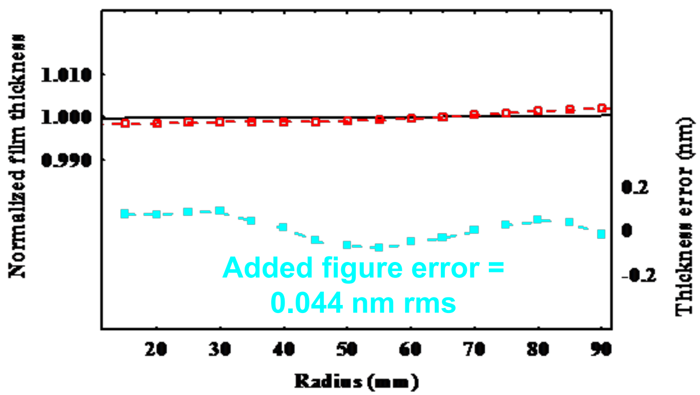


39-nm, 3:1 elbows *(Patrick Naulleau, LBNL)*

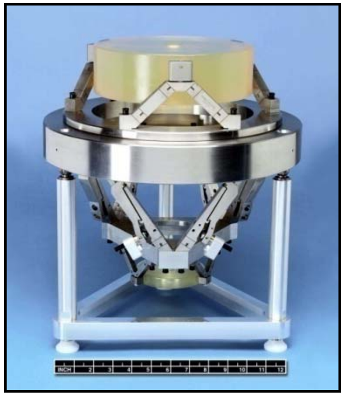


R. Soufli *et al*., *Proc. SPIE* 4343, 51-59 (2001)

M2 mirror, MET Set 1



MET camera

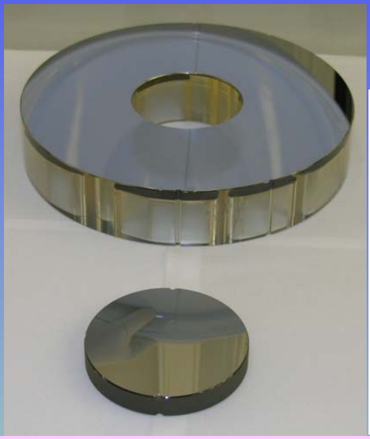


R. Soufli *et al*., *Appl. Opt.* 46, 3736-3746 (2007)

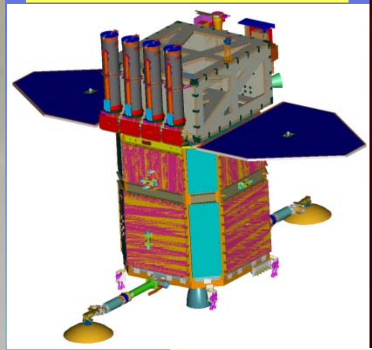
Measured wavefront = 0.55 nm rms
K. A. Goldberg et al, J. Vac. Sci. Technol. B 22(6), 2956-2961 (2005)

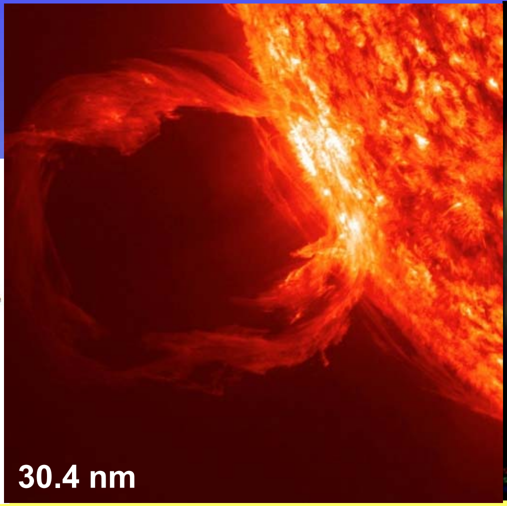
Printed 25 nm equal-line, and 29 nm isolated-line features
P. P. Naulleau et al, Proc. SPIE 5751, 56-63 (2005)

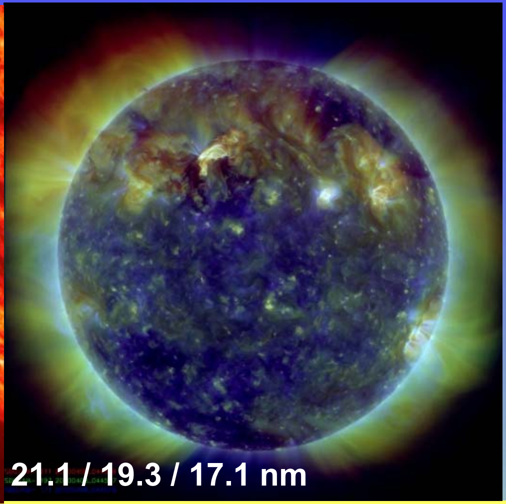
We have developed EUV multilayer optics and precision metrologies for next-generation EUV solar physics and space weather satellites



7 EUV wavelengths
(9.4 nm to 33.5 nm)

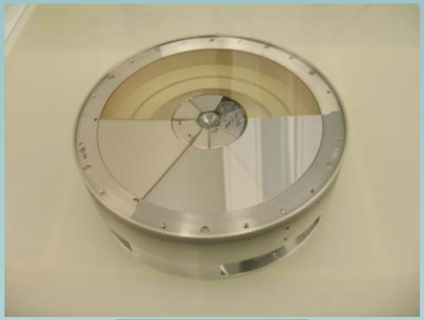


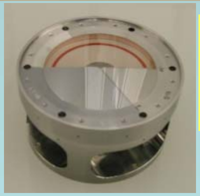


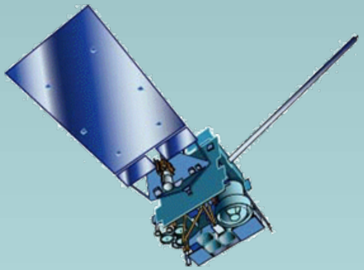


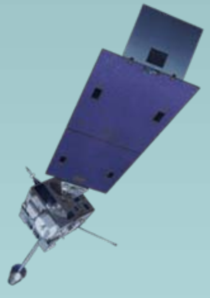
R. Soufli, *et al*, Appl. Opt. 46, 3156-3163 (2007).
R. Soufli, *et al*, Proc. SPIE 5901, 59010M (2005).

NASA's Solar Dynamics Observatory (SDO). Launch date: February 11, 2010.
http://sdo.gsfc.nasa.gov





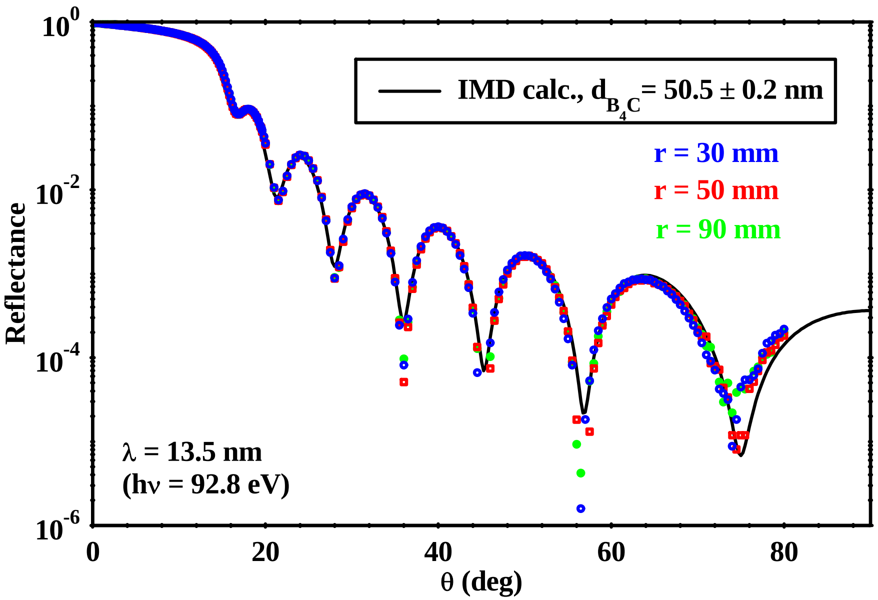


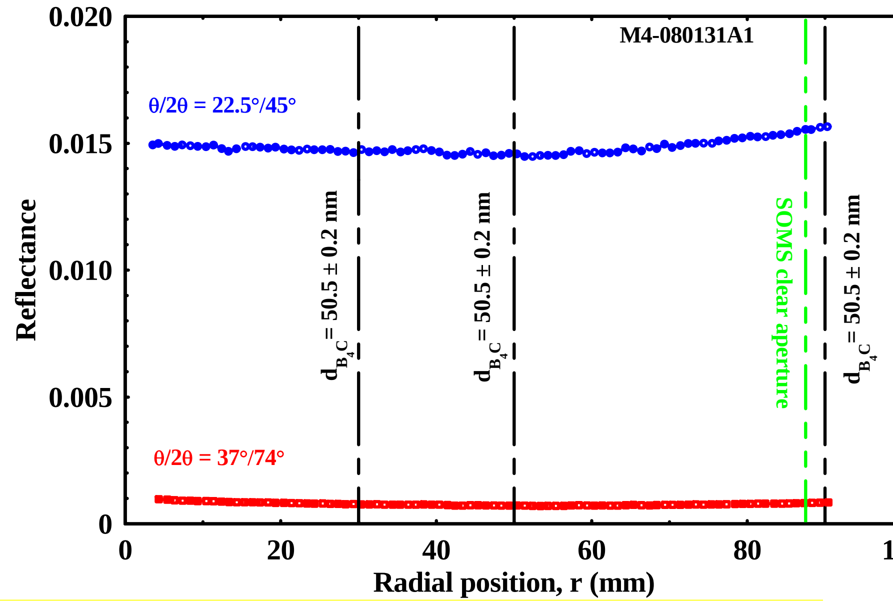


Multilayer-coated test mirrors for NASA/NOAA's GOES-R space weather satellite. 6 EUV wavelengths , 9.4 nm to 30.4 nm.
Launch date: 2014

SOMS and HOMS coating thickness uniformity is well within 1 nm rms specification

Measurements obtained at ALS beamline 6.3.2. (LBNL)





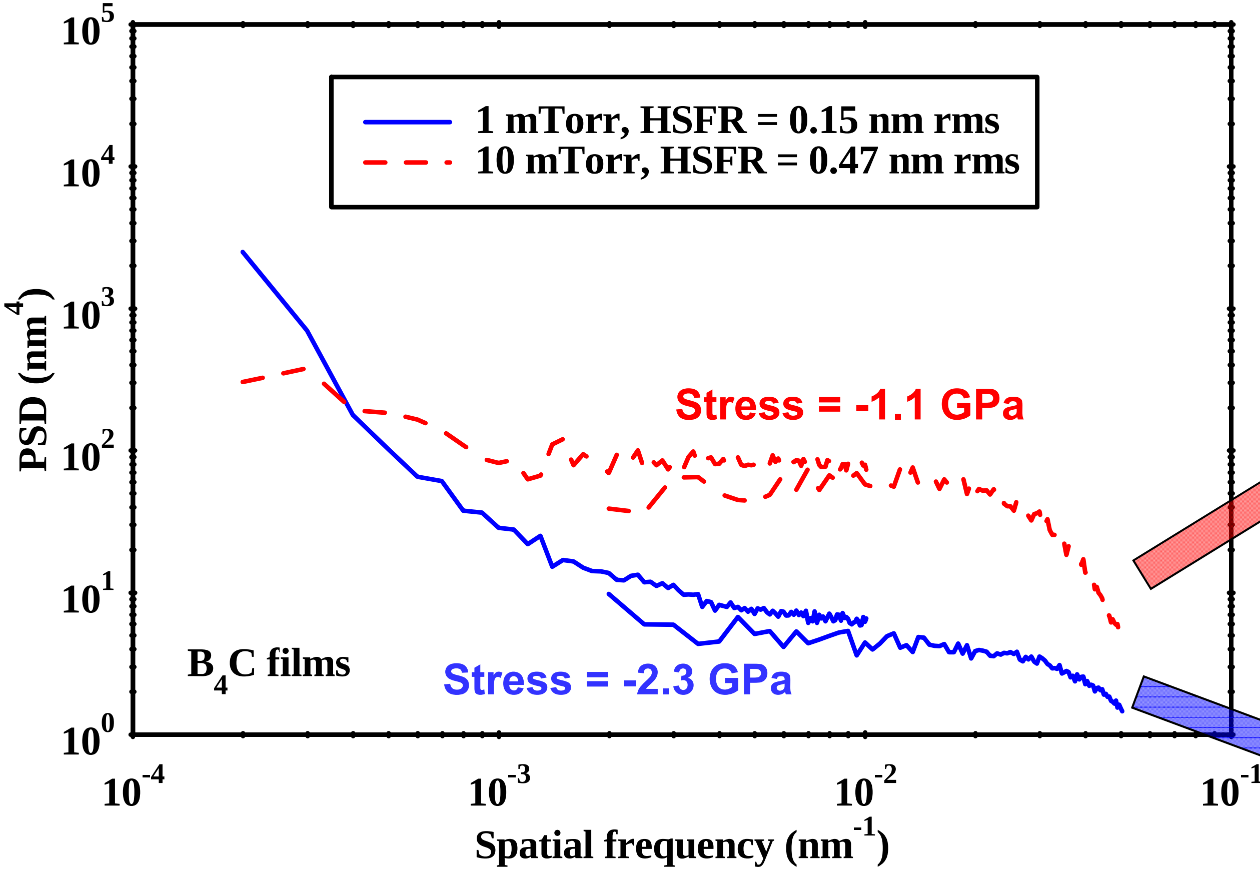
B_4C measured thickness variation: < 0.4 nm P-V, < 0.14 nm rms (< 0.28% rms) across the 175-mm SOMS clear aperture

SiC measured thickness variation: < 1 nm P-V, < 0.34 nm rms (< 0.7 % rms) across the 385-mm HOMS clear aperture

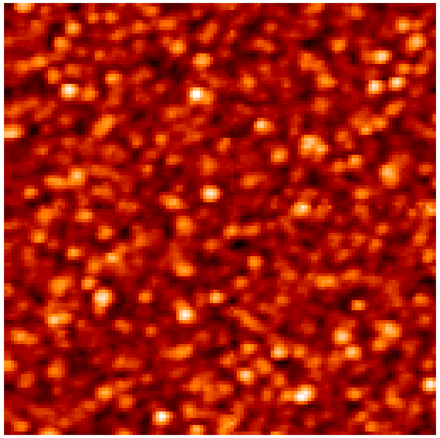
R. Soufli, M. J. Pivovaroff, S. L. Baker, J. C. Robinson, E. M. Gullikson, T. J. McCarville, P. M. Stefan, A. L. Aquila, J. Ayers, M. A. McKernan, R. M. Bionta, "Development, characterization and experimental performance of x-ray optics for the LCLS free-electron laser" Proc. SPIE 7077, 707716 (2008).

Regina Soufli
regina.soufli@llnl.gov

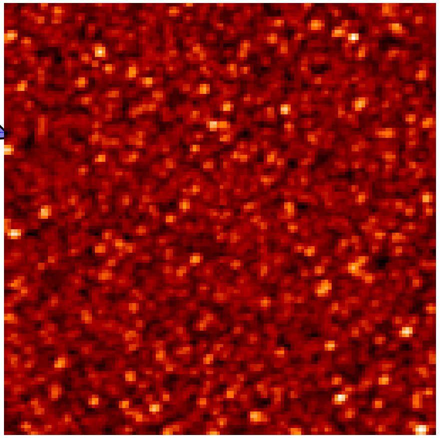
Boron carbide thin film development for LCLS SOMS mirrors



500×500 nm² detail from 2x2 μm² AFM scan



500×500 nm² detail from 2x2 μm² AFM scan



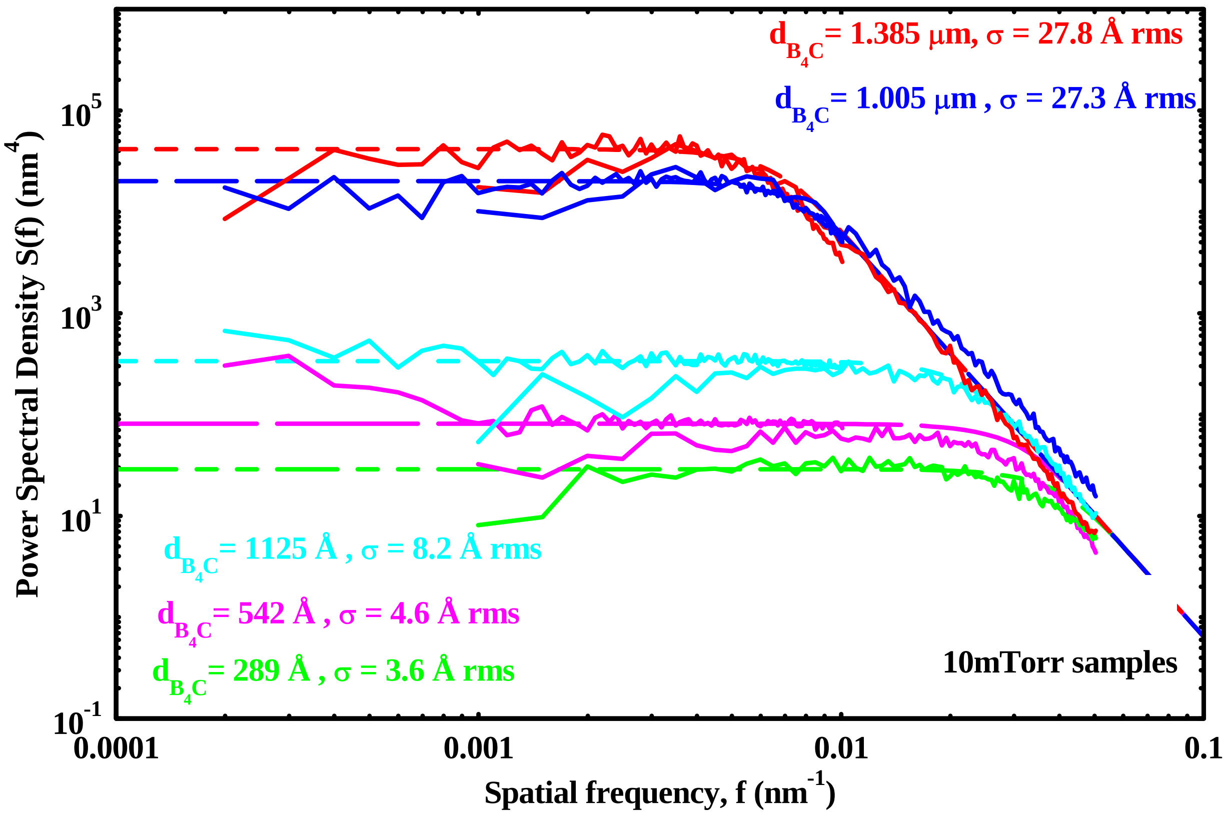
$$\sigma^2 = 2\pi \int_{f_1}^{f_2} f\, S(f)\, df$$

where $S(f) \equiv$ PSD (nm^4), $f_1 = 5\times10^{-4} nm^{-1}$
$f_2 = 5\times10^{-2}\ nm^{-1}$

R. Soufli, S. L. Baker, J. C. Robinson, E. M. Gullikson, T. J. McCarville, M. J. Pivovaroff, S. P. Hau-Riege, R. M. Bionta, “Morphology, microstructure, stress and damage properties of thin film coatings for the LCLS x-ray mirrors”, Proc. SPIE 7361, 73610U (2009).

Regina Soufli
regina.soufli@llnl.gov

AFM data from B_4C films of different thicknesses are fitted to obtain the stochastic growth model parameters



Fitted parameters: n, ν, Ω

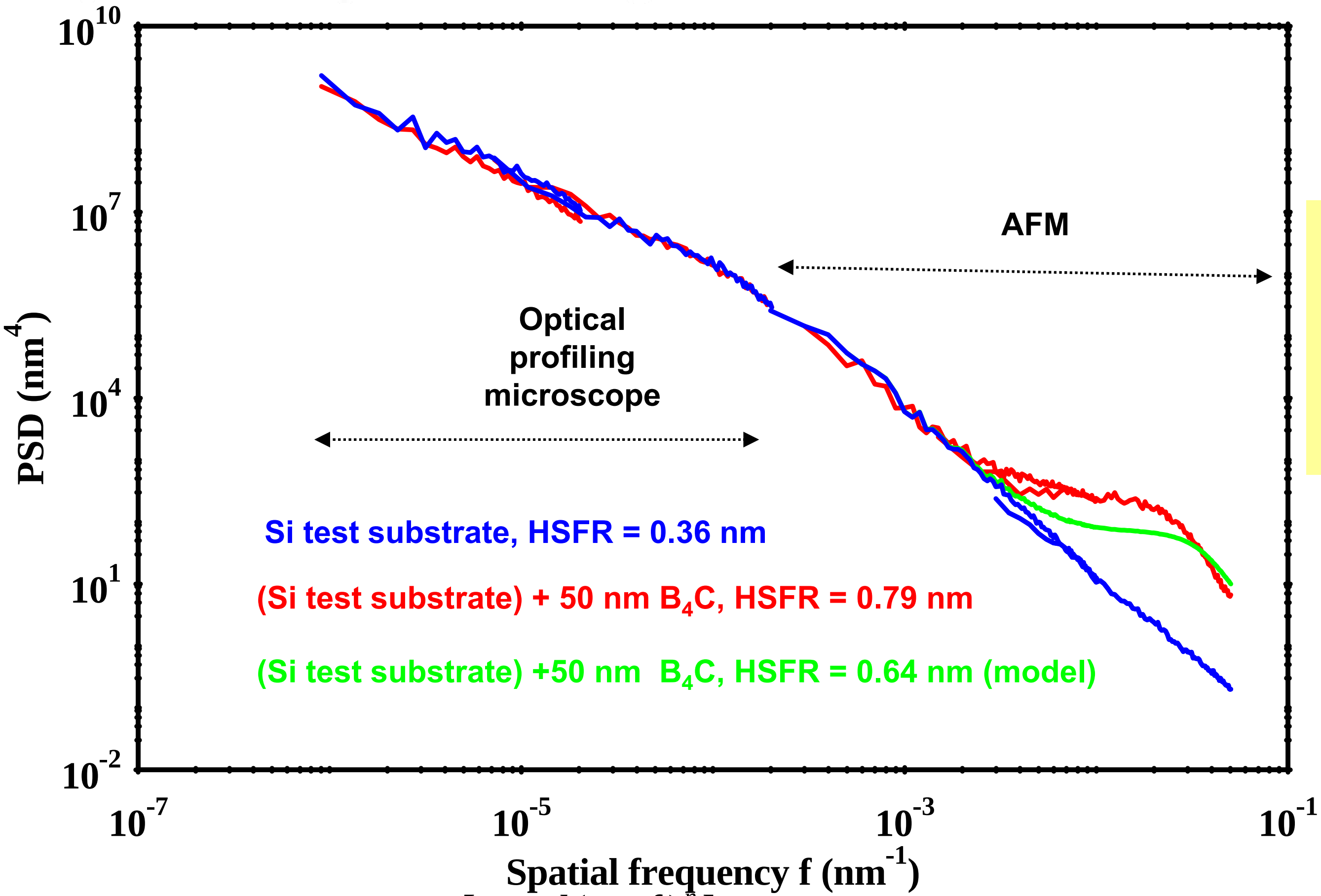
n = 4, ν / Ω = 5	
B_4C film thickness d_{B4C} (nm)	Ω (nm^3)
28.9	1
54.2	1.5
112.5	3
1005	20
1385	30

$$S^{B_4C\ film}(f) = \Omega \frac{1-\exp[-2\nu d(2\pi f)^n]}{2\nu (2\pi f)^n}$$

$$\sigma^2 = \int_{f_1}^{f_2} 2\pi f S^{B_4C\ film}(f) df$$ where $f_1 = 10^{-3}\ nm^{-1}$, $f_2 = 5\times 10^{-2}\ nm^{-1}$

Regina Soufli
regina.soufli@llnl.gov

We implemented a stochastic thin film growth model to predict B_4C film roughness on non-ideal substrates



R= 88.4 % (900 eV) at SOMS angle of incidence (0.79 deg), measured at ALS beamline 6.3.2., LBNL

$$S^{B_4C\ film}(f) = \Omega \frac{1-\exp[-2\nu d(2\pi f)^n]}{2\nu(2\pi f)^n}$$

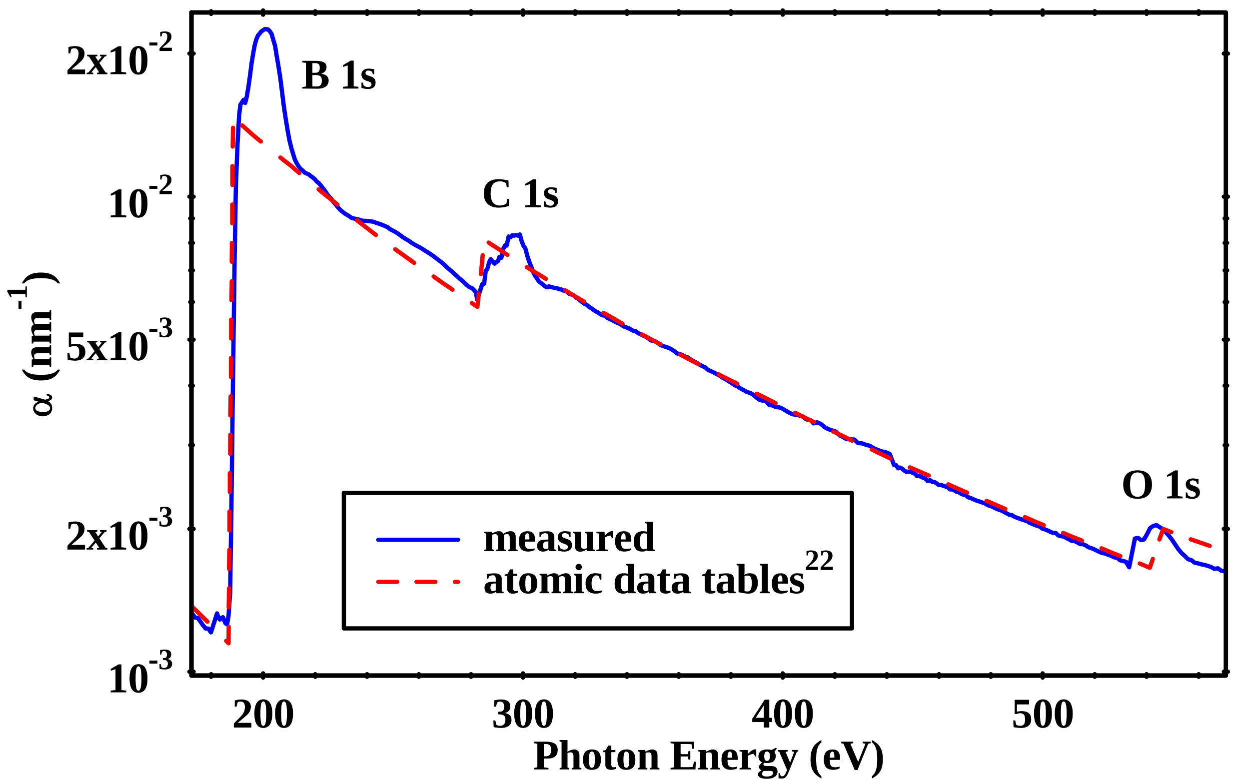
For d = 50 nm:
n=4, ν=7 nm^3 , Ω=1.4 nm^3

$$S(f)^{top} = S(f)^{B_4C\ film} + \{\exp[-\nu d(2\pi f)^n]\}^2 S(f)^{substrate}$$

Regina Soufli
regina.soufli@llnl.gov

Photoabsorption measurements yield updated values for the EUV/x-ray refractive index of B_4C films, including NEXAFS

Measurements obtained at ALS beamline 6.3.2. (LBNL)

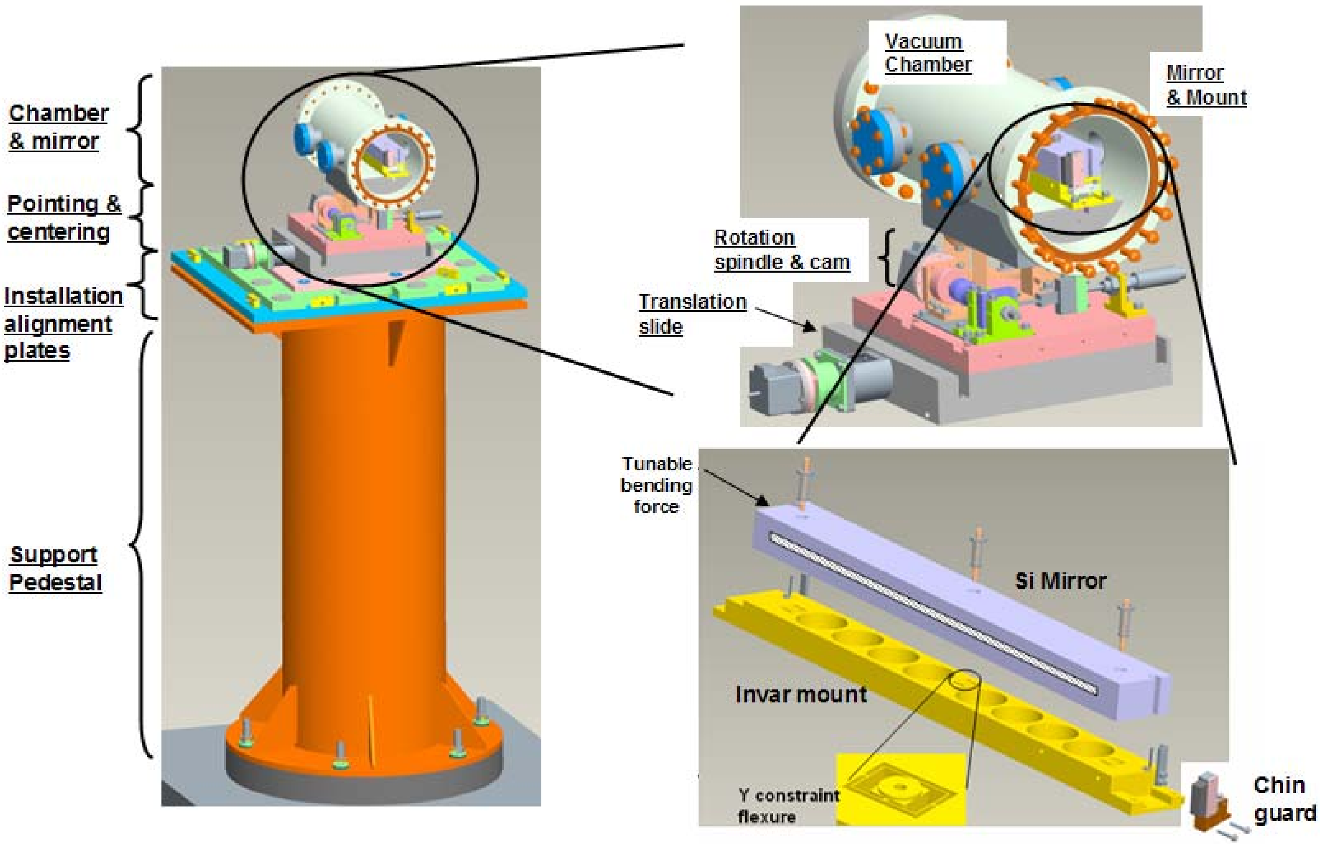


- **RBS results:**
  B=74%, C=20%, O = 6%
  B:C = 3.7
  ρ = 2.28 g/cm^3 (90% of bulk)
- **XPS results: top 9 nm of surface are O- and C-rich**

R. Soufli, A. L. Aquila, F. Salmassi, M. Fernández-Perea, E. M. Gullikson, "Optical constants of magnetron sputtered boron carbide thin films from photoabsorption data in the range 30 to 770 eV", Appl. Opt. 47, 4633-4639 (2008).

Regina Soufli
regina.soufli@llnl.gov

HOMS and SOMS installation and alignment at LCLS

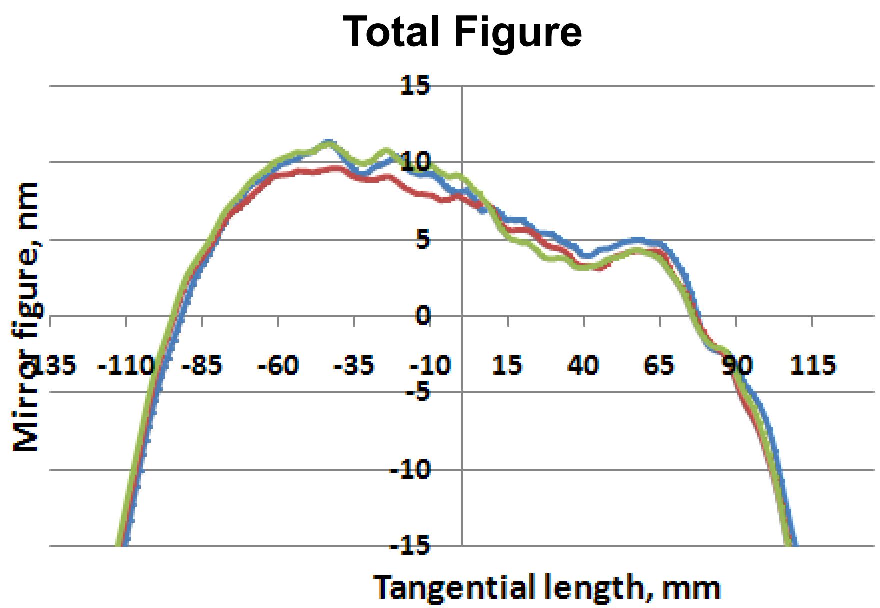


- T-controlled enclosure demonstrates ± 0.01 °C temperature and ± 30 nrad HOMS pointing stability
- HOMS figure can be remotely controlled

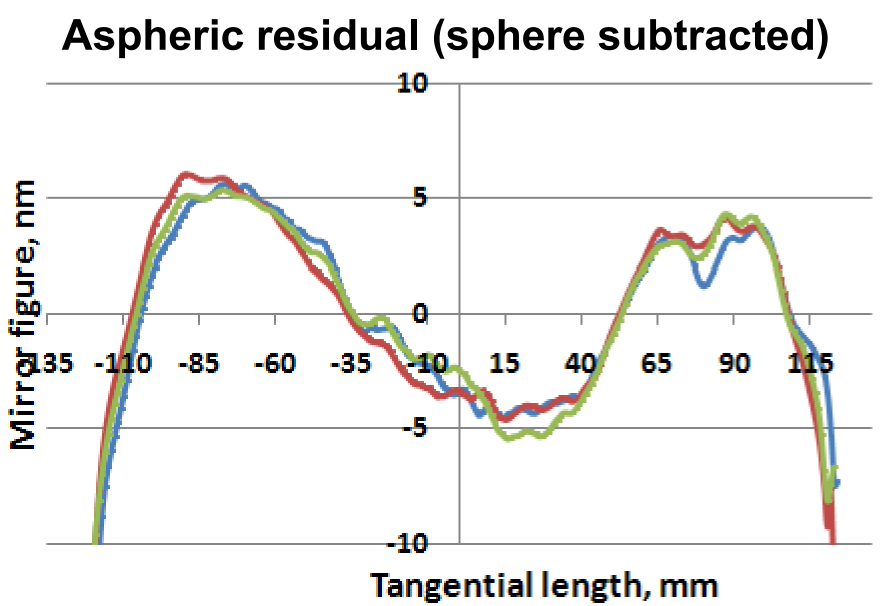
T. J. McCarville, P. M. Stefan, B. Woods, R. M. Bionta, R. Soufli, M. J. Pivovaroff, "Opto-mechanical design considerations for the Linac Coherent Light Source X-ray mirror system", Proc. SPIE 7077, 70770E (2008).

Regina Soufli
regina.soufli@llnl.gov

SOMS#1 figure summary



Before coating
Coated, un-mounted
Coated & mounted



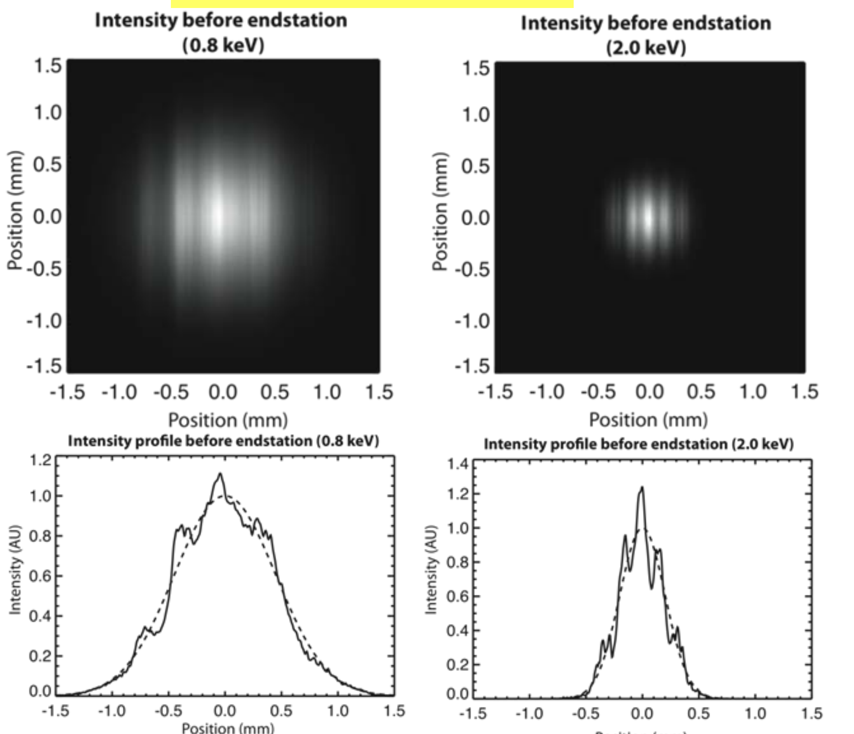
Before coating:1.81 nm & 0.19 μrad rms over central 200 mm
Coated, un-mounted:1.62 nm & 0.173 μrad rms
Coated & mounted:1.88 nm & 0.18 μrad rms

Regina Soufli
regina.soufli@llnl.gov

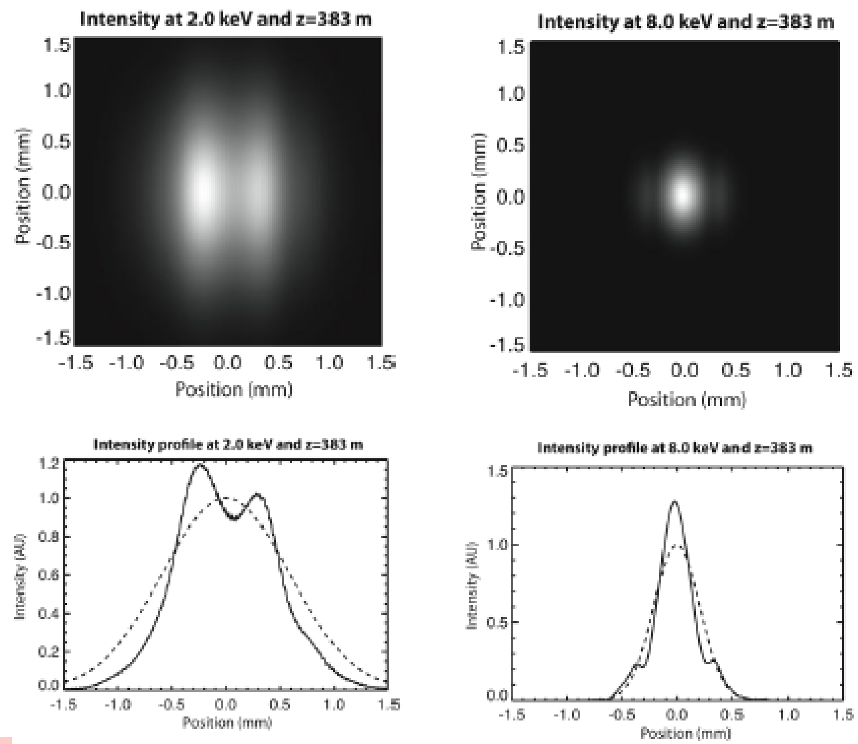
LLNL metrology data on SOMS and HOMS mirrors were used to predict LCLS coherent wavefront propagation and focal spot structure

- **Scalar diffraction model was employed**
- **Same methodology to select order of SOMS and HOMS elements for optimum performance**

SOMS branch line before AMO end station



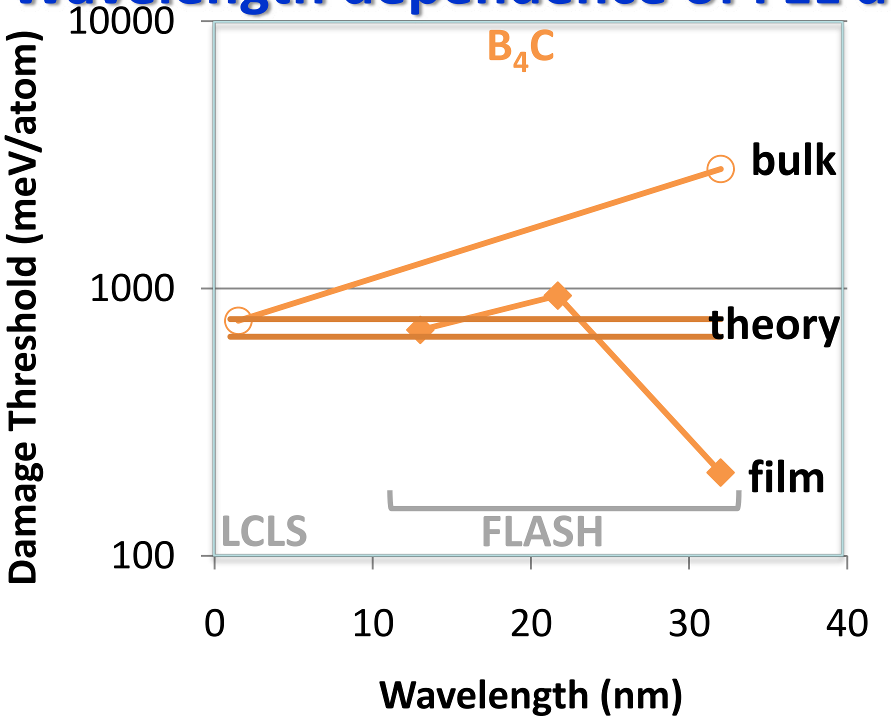
HOMS branch line before CXI end station

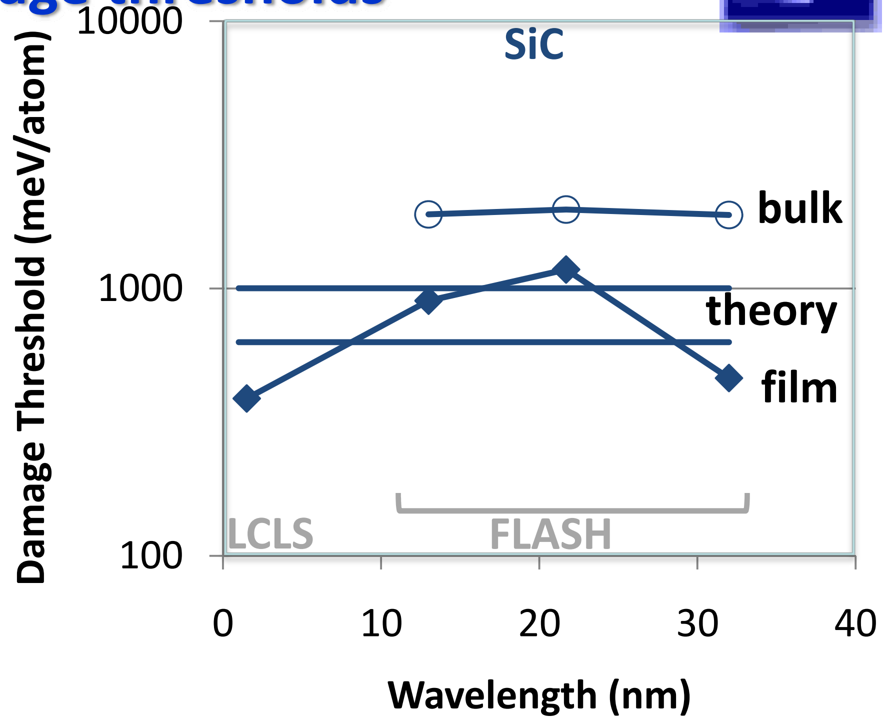


A. Barty, R. Soufli, T. McCarville, S. L. Baker, M. J. Pivovaroff, P. Stefan, and R. Bionta, "Predicting the coherent X-ray wavefront focal properties at the Linac Coherence Light Source (LCLS) X-ray free electron laser", Optics Express 17, 15508-15519 (2009).

Regina Soufli
regina.soufli@llnl.gov

Wavelength dependence of FEL damage thresholds



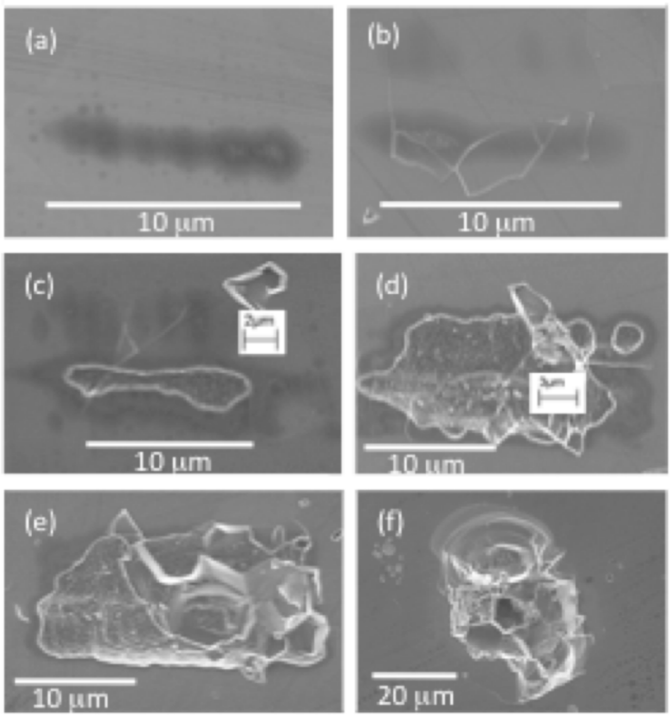


- **The single-pulse damage threshold is higher for bulk than for films**
- **Between 32 and 1.5 nm, the damage threshold is close to the melting threshold, in agreement with the general tenet for FEL optics design**

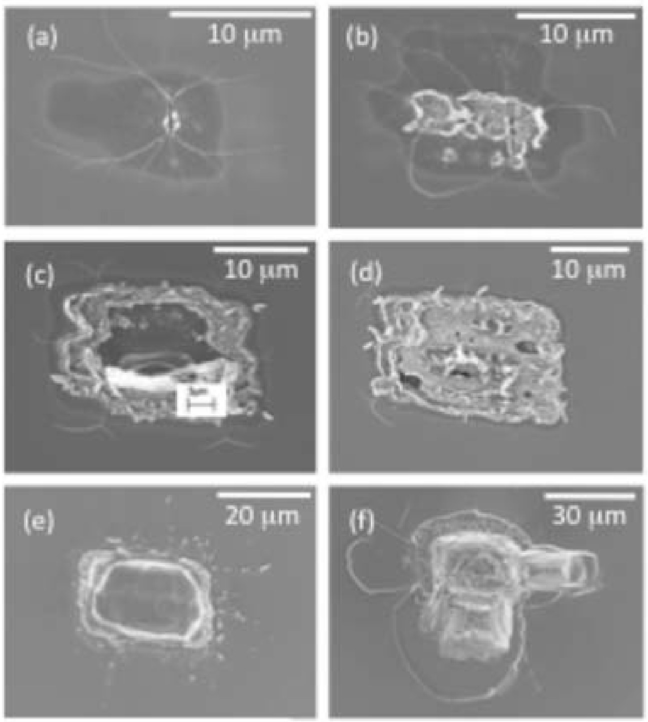
S. P. Hau-Riege *et al*, Interaction of low-Z inorganic solids with short x-ray pulses at the LCLS free-electron laser, submitted to Optics Express (2010).
S. P. Hau-Riege *et al*, "Wavelength dependence of the damage threshold of inorganic materials under extreme-ultraviolet free-electron-laser irradiation", Applied Physics Letters 95, 111104 (2009).
S. P. Hau-Riege *et al*, " Multiple pulse thermal damage thresholds of materials for x-ray free electron laser optics investigated with an ultraviolet laser", Applied Physics Letters 93, 201105 (2008).

Regina Soufli
regina.soufli@llnl.gov

LCLS FEL damage experiments at normal-incidence angles demonstrate ablation and cracking of materials



SEM pictures of bulk B_4C exposed to single XFEL pulses at 0.83 keV and peak fluences of (a) 2.6, (b) 5.4, (c) 7.0, (d) 11.4, (e) 19.6, and (f) 90.5 J/cm^2

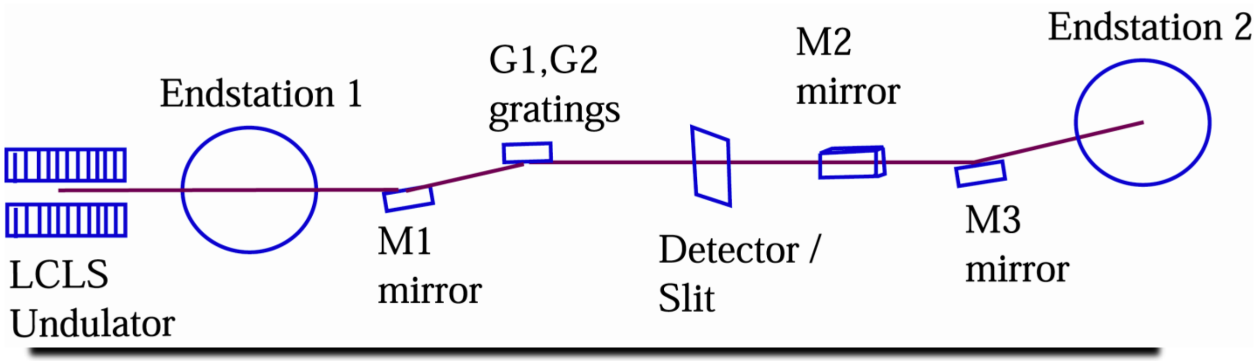


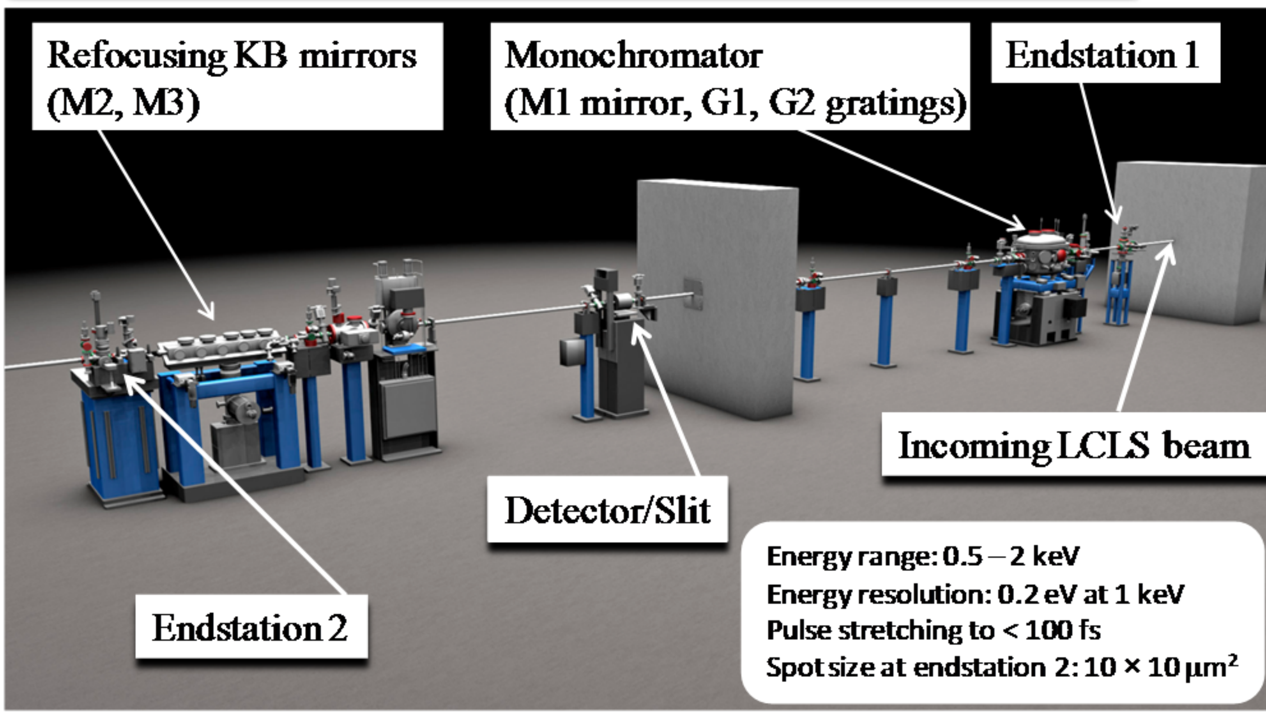
SEM pictures of a SiC film exposed to single XFEL pulses at 0.83 keV and peak fluences of (a) 1.0, (b) 1.6, (c) 2.9, (d) 5.8, (e) 14.8, and (f) 57.5 J/cm^2.

S. P. Hau-Riege *et al*, "Interaction of low-Z inorganic solids with short x-ray pulses at the LCLS free-electron laser", Optics Express (2010).

Regina Soufli
regina.soufli@llnl.gov

Soft X-Ray (SXR) materials science beamline at LCLS



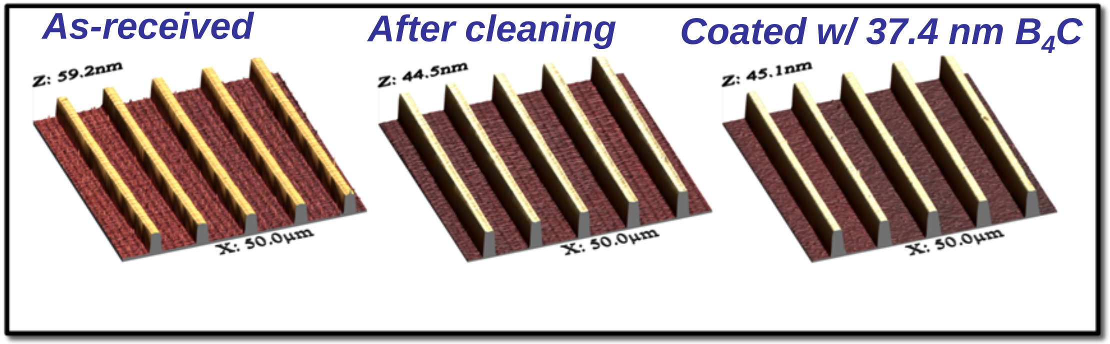


Regina Soufli
regina.soufli@llnl.gov

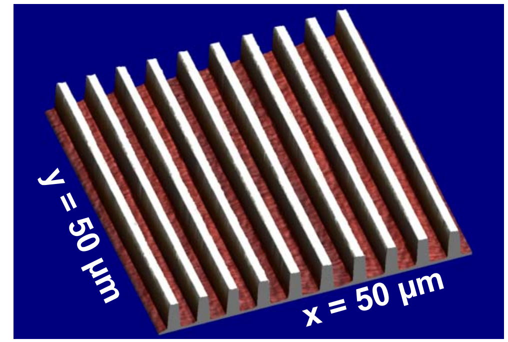
Monochromator gratings for LCLS SXR beamline

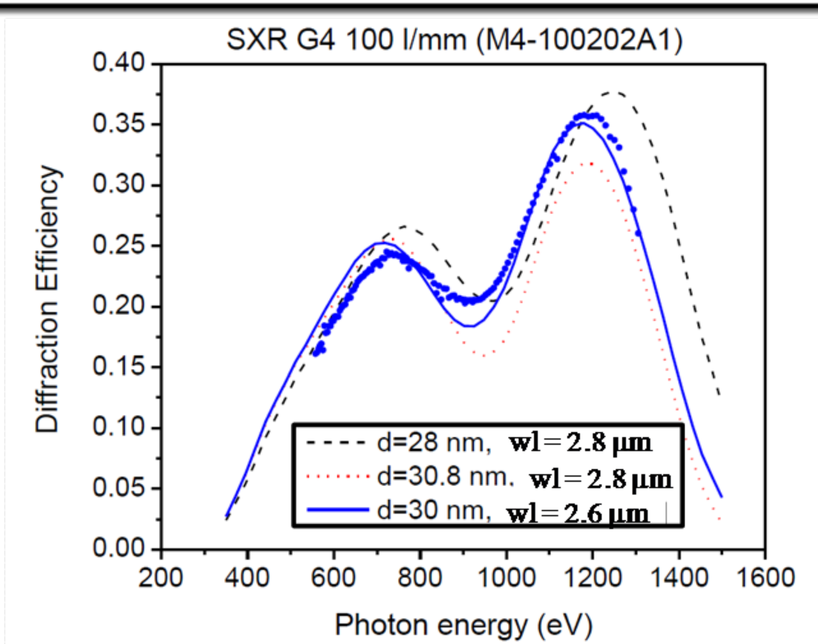
Grating substrate by Zeiss, ruling by Shimadzu

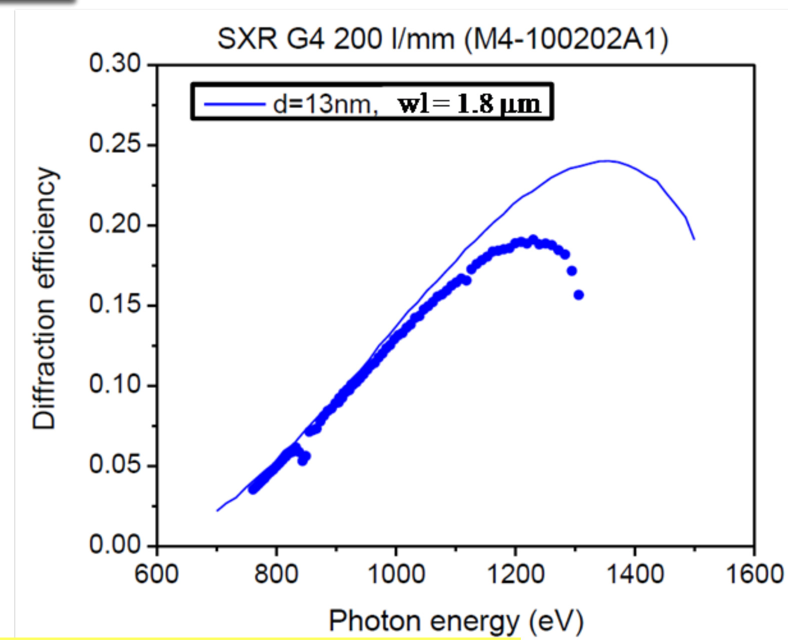
SXR test 100 l/mm grating



SXR G4 200 l/mm grating
After cleaning







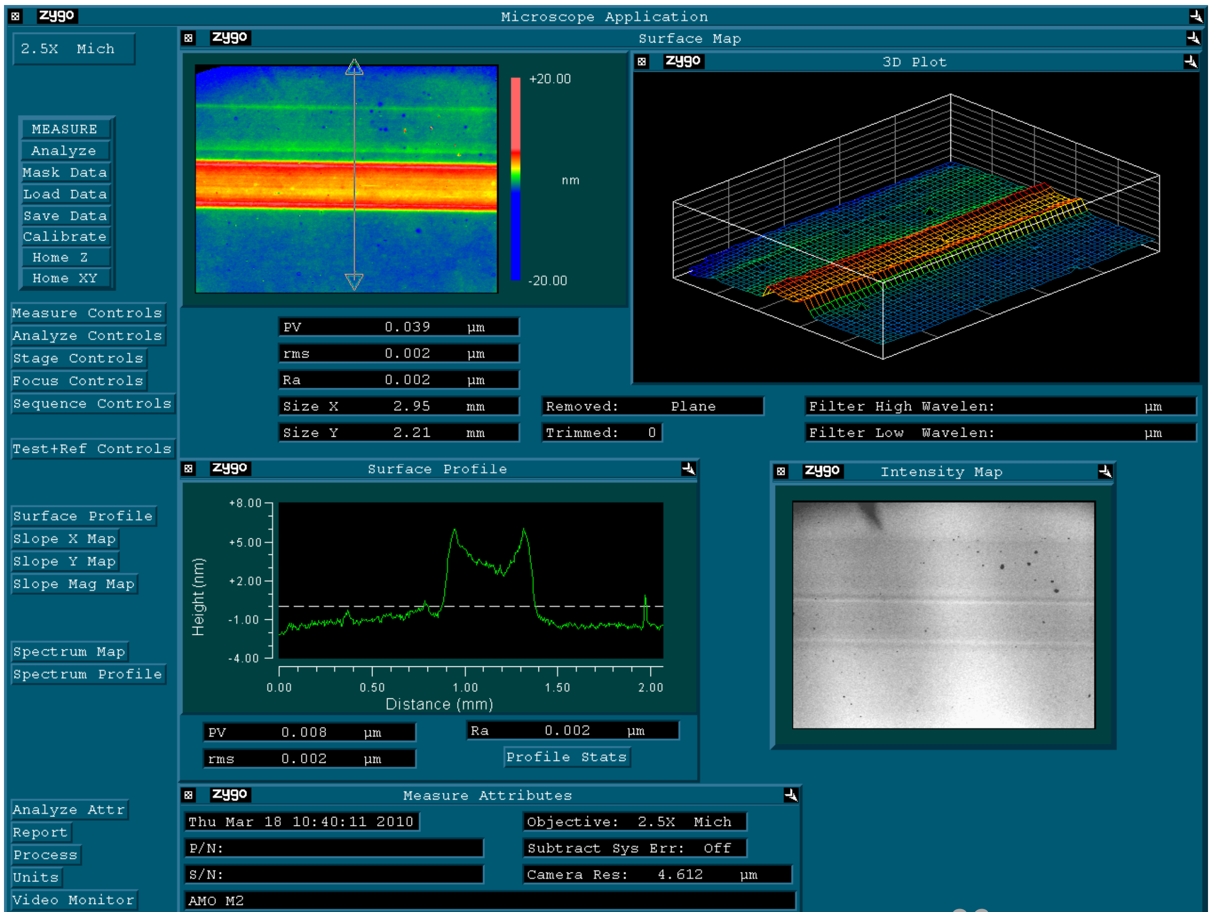
Measured at ALS beamline 6.3.2. Fits to measured data are preliminary

Regina Soufli
regina.soufli@llnl.gov

LCLS coating lifetime

- The FEL-exposed area on some of the LCLS x-ray mirrors has formed visible blemishes after several months of use
- These blemishes is currently attributed to imperfect vacuum conditions combined with the very high intensity of the LCLS FEL beam

AMO beamline M2 K-B mirror



- Contact profilometry measurements on a witness piece revealed a blemish height of 15 to 38 nm.
- XPS on blemish area: 87% C, 12% O and 1% Si. C found both as hydrocarbon and graphite

Regina Soufli
regina.soufli@llnl.gov

Future work: lifetime improvement of LCLS coatings

Challenges:

- **Cleaning/recovery techniques must preserve the coating composition, thickness, uniformity, etc so that the x-ray reflectance, beam coherence and damage thresholds are also preserved.**
- **Carbon, the major component of the blemish regions, is also a constituent of the B_4C and SiC coatings.**

Candidate recovery strategies for coated mirrors/gratings:

- **UV-ozone cleaning: Preliminary experiments on B_4C thin films show degradation of coating performance (through oxidation and/or roughening)**
- **Plasma (glow discharge) cleaning techniques**
- **Substrate recovery and re-coating**

Regina Soufli
regina.soufli@llnl.gov

Summary and future plans for LCLS x-ray optics at LLNL

450 mm

HOMS mirrors, coated with 50 nm SiC

- Developed highly reflective B_4C and SiC coatings for the LCLS x-ray mirror systems that preserve the LCLS wavefront
- Designed, developed, characterized and installed x-ray mirrors for the LCLS SOMS and HOMS systems and LCLS AMO and SXR beamlines
- SiC-coated X-ray mirrors for CXI and MEC beamlines are currently underwa
- LLNL precision metrology results on SOMS and HOMS mirrors were incorporated in coherent wavefront propagation models, to predict LCLS focal spot structure
- Damage experiments are being performed on B_4C and SiC thin films and bulk samples
- **Future work:** investigate damage and contamination mechanisms on x-ray optics under LCLS FEL irradiation, improve x-ray optics lifetime.

Regina Soufli
regina.soufli@llnl.gov

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- LCLS executive management at SLAC
- Angela Craig, Bruce Rothman and Patrick Schnabel (Evans Analytical Group, Sunnyvale, California) for the XPS and RBS measurements
- This work performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344. Work was supported in part by DOE Contract DE-AC02-76SF00515. This work was performed in support of the LCLS project at SLAC.

Regina Soufli
regina.soufli@llnl.gov